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SEPTEMBER 29, 1941
VOLUME 30, No. 20

AMERICAN DYESTUFF REPORTER

OFFICIAL PUBLICATION
of the
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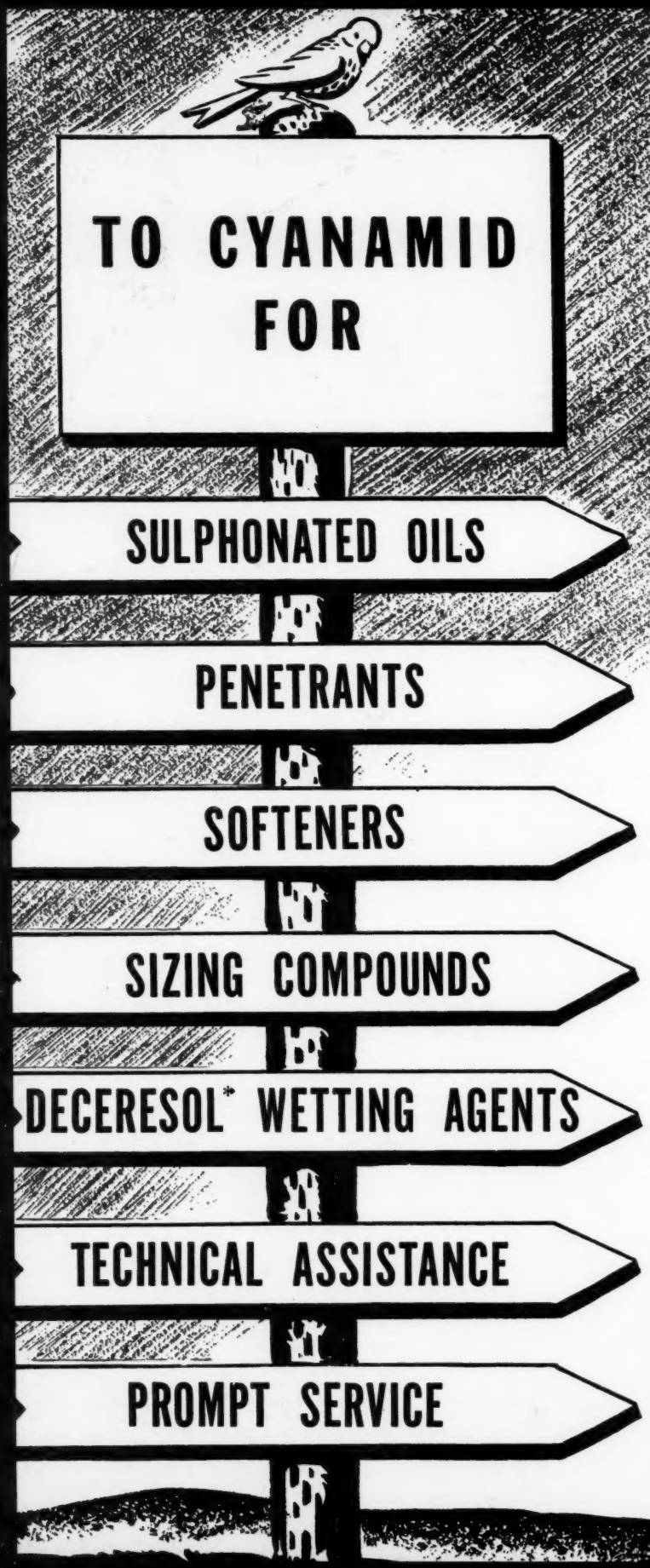

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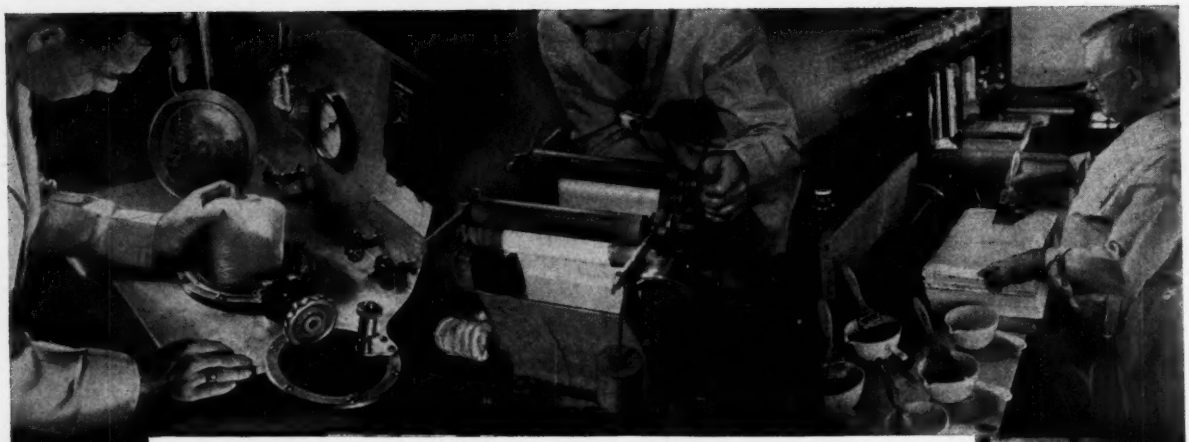

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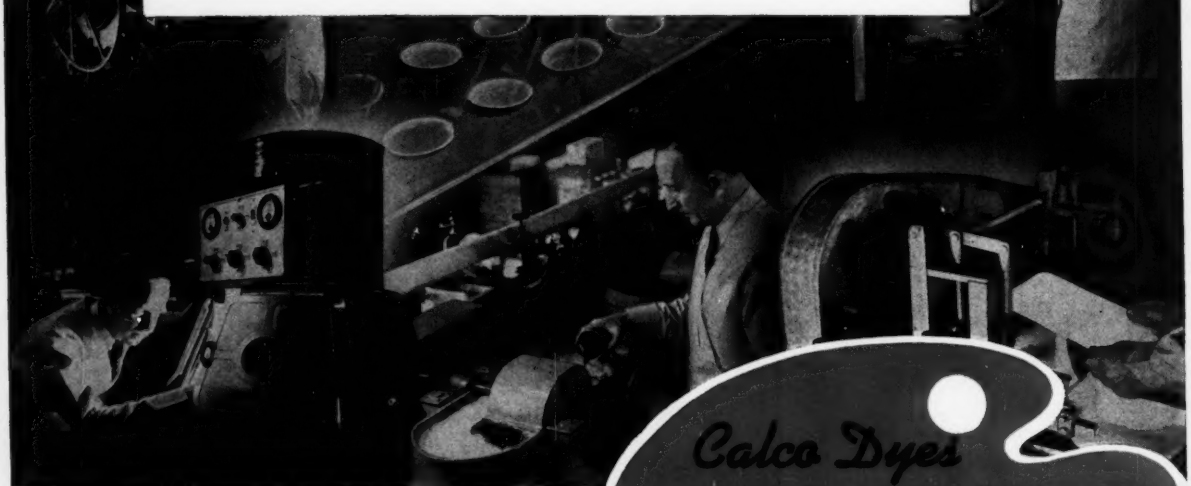

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DYESTUFF REPORTER

VOLUME 30 SEPTEMBER 29, 1941 NUMBER 20

DYES FROM CITRAZINIC ACID

NICHOLAS L. KALMAN*

MOST organic chemists when asked about the formula of citrazinic acid would probably be unable to answer the question. No wonder, as the Chemical Abstracts in its Indexes for the last 33 years lists a total of only 2 references about this product. There are about thirty publications discussing and referring to citrazinic acid but twenty-five of these were published before 1907. In fact no economically feasible method for its preparation has been described in the literature since 1894, nearly half a century ago. From this, citrazinic acid might appear to be a chemical curiosity of little commercial promise. Recently, however, methods of preparation have been brought up-to-date and somewhat improved in efficiency. While the acid is not now being manufactured, it has been made on a pilot plant scale. If there should develop a sufficient large-scale demand to justify production, it is possible that citrazinic acid could be made generally available at a price competitive with other chemicals of the same general properties.

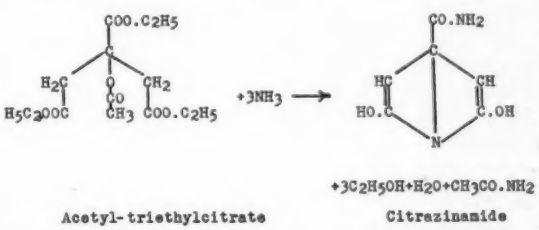
Citrazinic acid was discovered by I. Behrmann and A. W. Hofmann[1] who obtained it by the evaporation to dryness of an aqueous solution of citramide or one of the citramic acids, with HCl. The new compound which the authors named citrazinic acid had a formula of $C_6H_5NO_4$. The yield obtained by this process was only 5 per cent of the amide used.

Much better yields have been obtained by dissolving an amide of citric acid, such as citramide, in 70-75 per cent H_2SO_4, heating to 130° C. to achieve solubilization, cooling, and then pouring the reaction mixture into cold water, when citrazinic acid precipitates in a practically pure form.

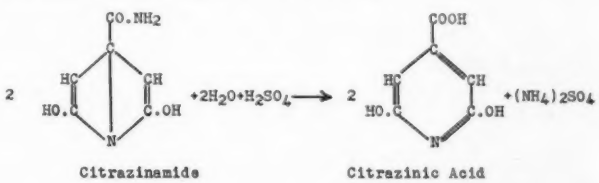
It has been stated that it was not necessary to isolate the amide of citric acid, but one may start with the tri-methyl ester of citric or acetyl-citric acid which are treated with concentrated ammonia. After evaporation, the resulting mass is reacted with H_2SO_4, and subsequently poured into water, as described above. In such a manner, the yield reaches 25 per cent of the ester employed (38 per cent of the theoretical).

Recent discoveries and improvements raised this yield still much higher, thereby enabling the manufacture of citrazinic acid and its derivatives at moderate costs.

The transformation from acetyl triethylcitrate to citrazinamide (which material is the precursor of citrazinic acid) may be visualized in the following manner:



The subsequent decompositions of citrazinamide to citrazinic acid takes place as follows:



Citrazinic acid, as it may be seen, is a derivative of pyridine, having two phenolic groups in the 2 and 6 positions and a carboxyl group in the 4 position. As pyridine-4-carboxylic acid is called isonicotinic acid, citrazinic acid may be termed as 2:6-dihydroxy-isonicotinic acid.

Citrazinic acid was prepared not only from the above, but also from citric acid[6], esters of citric acid[5], acetanhydrocitric acid[5], acetyl-triethylcitrate[2, 4], anhydroaconitic acid[5], amides of aconitic acid[4], esters of aconitic acid[3, 4], ∝-cyantriethyl aconitate[10]; in all these instances the reaction mechanism was basically the same. Other preparations include those from 2.6-dichloro-isonicotinic acid[8], 2.6-dioxy-pyridine-dicarboxylic acid-(3.4)-diethyl ester[9, 10], and finally citrazinamide[2].

As it was shown, citrazinic acid may be prepared, as a rule, from suitable derivatives of citric acid, with the help of ammonia, the aliphatic starting out materials becoming cyclicized in the process.

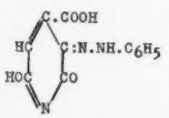
Citrazinic acid is a yellow powder which chars without melting over 300° C. It is very difficultly soluble in hot water, difficultly soluble in hot concentrated HCl and warm concentrated H_2SO_4, slightly soluble in formic acid and very slightly soluble in glacial acetic acid. It is readily soluble in alkalies and alkali carbonates, soluble in pyridine

*Arthur D. Little, Inc.

and readily soluble in quinoline but the two last solutions are very dark. When citrazinic acid is dissolved in an aqueous Na_2CO_3 solution, and then acetic acid is added, citrazinic acid remains in solution. Aqueous triethylamine is a solvent, while the anhydrous base is not. Citrazinic acid is insoluble in neutral organic solvents; ethylene glycol was the only one of the numerous solvents which have been tried which dissolved some citrazinic acid.

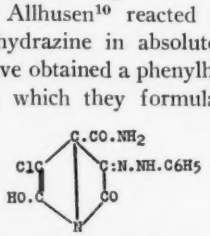
Due to its configuration, citrazinic acid may be utilized as an intermediate for dyestuff preparations. Furthermore, about sixty derivatives of citrazinic acid have been prepared and described in the literature, and a number of these present interesting possibilities as intermediates in the dyestuff field. Theoretical considerations of these products reveal that various reactions should enable the preparation of widely varied dyes belonging in divers classes. The literature mentions only two instances in which reactions coming under these headings were performed.

Sell and Easterfield[8] dissolved citrazinic acid (1 mol.) in an excess of a caustic soda solution, and then it was slightly acidified with acetic acid. To this an ice cold solution containing diazo-benzene-chloride (1 mol.) and some free hydrochloric acid were added. A bright yellow precipitate was immediately formed, and this, when recrystallized from water, proved to be the acid sodium salt of the hydrazo-acid. The authors postulated that the constitution of the hydrazo-acid was most simply expressed by the formula:

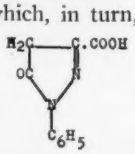


Such a substance should, however, easily yield a pyrazolone by elimination of the elements of water but the authors have not succeeded in effecting this condensation.

Ruhemann and Allhusen[10] reacted trichloro-citrazinamide with phenylhydrazine in absolute alcohol, and reported that they have obtained a phenylhydrazone of monochlorcitrazinamide, which they formulated as follows:



This compound is really 3-phenylazo-5-chloro-citrazinamide, and possesses the structure of an azo dye. On refluxing it with hydrochloric acid, scission has taken place, the product being transformed into 1-phenyl-3-carboxy-pyrazolone-(5) which, in turn, couples readily with



diazonium bases to form dyestuffs. The above products, at least as far as the record goes, have not been examined for their dyeing properties.

The phenolic groups of citrazinic acid enable its use as a coupling agent in the azoic dye field, and we have investigated some of the inherent possibilities of using citrazinic acid and some of its closest derivatives as coupling agents with diazo compounds. We have found that citrazinic acid couples readily, not only once but twice. Furthermore, citrazinic acid possesses a configuration which responds well to a chroming treatment, and the top chrome dyes so obtained have been also examined.

PREPARATION OF AZO DYES USING CITRAZINIC ACID AS COUPLING AGENT

Preparation of Diazo Solution

The amines used were either dissolved in aqueous HCl solution or were present in a finely divided form in cases where they were insoluble. There were always present 2½ moles of HCl per amino group. The amines were diazotized by the slow addition of sodium nitrite solution, during which time the temperature was always maintained below 10° C. The reaction required from 20 to 30 minutes; it was over when a slight excess nitrous acid persisted for 15 minutes when tested with starch-iodide paper. The slight excess of nitrous acid was then destroyed by the addition of urea or sulfamic acid.

The end points of the diazo reaction of the various amines, as determined by the starch-iodide test, were sharp in all cases, except with sulfanilic acid, which persisted in giving a slight blue coloration to the test paper, even in the presence of an excess of amine.

Coupling and Isolation of Dyes

The couplings were carried out in baths of various pH values. Solutions of sodium hydroxide, sodium carbonate, and sodium acetate—acetic acid buffers were used. depending on the nature of the coupling components and on the dyestuff to be prepared. The citrazinic acid or derivative was dissolved in the appropriate solution and the diazo solution was added within about ten minutes with constant stirring. The temperature of the coupling solution varied with the different diazo compounds used, according to their stability. For example, in the case of aniline the solution was maintained in the vicinity of 0° C., while in the case of benzidine room temperature was satisfactory. The solutions were then allowed to come to room temperature and to stand, with stirring, for varying lengths of time for the reaction to become complete. The duration of this time interval was governed by the reactivity of the diazo salt, ranging from 1-2 hours, generally, to many hours in the case of the second benzidine coupling. In most preparations the solutions were worked up from this point, but in others the solutions were heated to 60-70° C. in order that the reaction become complete.

The end points of the couplings were determined by spotting the solutions on filter paper.

As a rule, it becomes necessary to salt out the dyes from solution by the addition of NaCl when the mixture is filtered. Great difficulty has been experienced in filtering some of these solutions because of their colloidal nature; heating the mixture to 60-70° C. and allowing it to stand many hours to agglomerate the particles have proved helpful. Wherever possible, the dyes were isolated from

alkaline solutions in order to obtain the dye in its most soluble form, as the mono- or polysodium salts. Where this has been found impossible because of the great solubility of the dye, precipitation was aided by the careful addition of acetic acid. The dyes were well washed with salt solution and the filter-cakes sucked dry with the aid of a rubber dam. The dye-filter cakes were dried in a vacuum oven at 60° C., and powdered.

Because citrazinic acid is capable of coupling twice and readily in each position, we have generally utilized a weakly acid coupling medium in the preparation of monazo dyes. This was done to prevent the formation of any disazo dye, that might have formed in an alkaline solution where the coupling activity is supposedly far greater. Sodium acetate was used as a buffer to maintain the solution weakly acid to congo-red paper. Where two identical diazo groups were coupled, a sodium hydroxide or carbonate solution was employed.

In general, the technical grades of various intermediates were used in the preparation of these dyestuffs. Citrazinic acid and its methyl and ethyl ester were furnished by Charles Pfizer & Co., and the butyl ether of citrazinic acid was prepared by saponification of the butyl-ether-ester that was submitted by Pfizer.

DYEING PROCEDURES WITH CITRAZINIC DYES

Testing of Citrazinic Azoic Dyes

The dyes prepared were dyed on wool skeins both as acid colors and top chrome colors.

Procedure for Acid Colors

The dye bath was made up using 2 per cent dye, 10 per cent Glauber's salt, and 3 per cent acetic acid, all based on the weight of wool to be dyed. Volume of the bath was 50:1.

The wool was scoured, washed and wet out thoroughly and entered at 70° F. Brought to the boil in 20 minutes and boiled for ½ hr. Exhausted with

3% acetic acid for light colors (yellows and oranges) or

2% sulfuric acid for dark colors (browns, reds and blacks)

Boiled for an additional ½ hr. Removed from bath, rinsed well, and dried.

Procedure for Top-Chrome Colors

The dyeing was carried out as described above. The dye-bath was cooled slightly and 2 per cent of sodium bichromate was added based on the weight of wool. Boiled for ½ hr., removed from bath, rinsed well, and dried.

CHARACTERISTICS OF DYEINGS WITH CITRAZINIC DYES

Fastness

The dyed skeins have been tested for light fastness with a Fade-o-meter. The ratings in the table below are according to the du Pont Scale, the light fastness as indicated by Fade-o-meter test being:

1. Only a trace of change after 150 hrs' exposure.
1-2. Noticeable change between 100-150 hrs.
2. Noticeable change between 50-100 hrs.
3. Noticeable change on 25 hrs'. exposure.
4. Noticeable change on 10 hrs'. exposure.
5. Noticeable change on 5 hrs'. exposure.

The washing tests were made in a neutral ½ per cent soap solution at 120° F., the ratings meaning the following:

1—Very good
2—Good
3—Fairly good
4—Moderate
5—Poor

The change of shade on washing is designated as follows:

B—Bluer
G—Greener
R—Redder

Shades and Brightness

In order to visualize the shades of the dyes prepared from citrazinic acid and its mono-butyl ether, we have tried to match the shade of these citrazinic dyes as closely as possible with some dyes manufactured by Du Pont de Nemours & Co. or National Aniline & Chemical Co. but in some instances we did not find any dyes which were identical or fairly close to our shades. This is especially true regarding some of the citrazinic chrome colors.

The following tabulations of the various dyes prepared and of their characteristics will throw some light on the subject of simple azo dyes preparable from citrazinic acid and its mono butyl ether derivative. In the column "strength" we only noted apparent deviations between the strengths of the citrazinic dyes and the other dyes with which they have been compared. It should be noted that these comparisons were very crude and should be considered only as guides. Some of the fastness tests have been omitted for one reason or the other.

One of the outstanding features of these dyes is the uniform brightness of the chrome colors but a considerable number of them have comparatively weak tinctorial strength. Another advantageous characteristic is the good light-fastness of a number of these dyes; this light-fastness seems to be due, at least to a considerable extent, to the citrazinic acid molecule. It is interesting to note the fact that while Resorcinol Yellow is very fugitive to light, the analogous citrazinic acid dye has good light fastness. Our results indicate that when a carboxyl group is in an ortho position, or a sulfonic group in an ortho or para position to the amino group, in the diazo component, the resulting citrazinic dyes have good or excellent light fastness. Still another important feature of these colors is that they possess good fastness to dry-cleaning solvents, such as Stoddard solvent, carbon tetrachloride, perchlorethylene and trichlorethylene.

On the other hand, the washing fastness tests have shown that all the dyes made from citrazinic acid bled very badly, while a few made with the butyl-ether bled but slightly. The washing fastness of the top chromed

Monoazo Dyes Made With Citrazinic Acid

Dyed as Acid Colors

No. of Dye	Diazo Component	Light Fastness	Washing Fastness	Shade	Strength
1	Aniline	2	5	Nearest to Pontacyl Light Yellow 3G	
2	∝- Naphthylamine	2-3	5	Similar to du Pont Orange G, but considerably duller	
3	o-Anisidine	2-3	5	Similar to du Pont Neutral Yellow GS, but brighter	
4	p-Amino-acetanilide			Similar to du Pont Milling Yellow R	
5	2.5-Dichloraniline	2-3	5	Similar to Pontacyl Light Yellow GS, but slightly greener	Little weaker
6	p-Nitraniline	4	5 R	Dull mustard yellow, duller than du Pont or National colors	
7	Picramic acid	1	5	Dark red, somewhat like National Erie Fast Red FD, but yellower	
8	Anthranilic acid	1	5	Similar to National Fast Wool Yellow 3 GL, but slightly brighter and yellower	
9	p-Aminobenzoic acid	3	5	Similar to Pontacyl Light Yellow GS	
10	Sulfanilic acid	2	5 R	Similar to Quinoline Yellow, but slightly redder	
11	Metanilic acid	2-3	5	Near to Pontacyl Yellow GX, but duller	
12	Naphthionic acid	1-2	5	Nearest to Metanil Yellow conc.	
13	2-Naphthylamine-1-sulfonic acid	1-2	4 G	Yellowish orange	
14	2-Naphthylamine-6-sulfonic acid	2	4	Similar to du Pont Neutral Yellow GS	1/2 Strength
15	2-Naphthylamine-7-sulfonic acid	2-3	5	Similar to du Pont Neutral Yellow GS, but brighter	
16	1-Naphthylamine-5-sulfonic acid	2	5	Similar to du Pont Milling Yellow R conc.	
17	1-Naphthylamine-8-sulfonic acid	3	5	Similar to du Pont Milling Yellow R, but much duller and not as red	
18	p-Nitraniline-o-sulfonic acid			Dull yellow, duller than du Pont or National colors	
19	o-Aminophenol-p-sulfonic acid	3	5 R	Brownish yellow; different from du Pont or National colors	
20	1-Amino-2-naphthol-4-sulfonic acid	3	5 B	Somewhat like du Pont Milling Red B, but duller and yellower	Weaker
21	Aminoazobenzene-p-sulfonic acid	1-2	4	Similar to du Pont Orange G, but duller and redder	
22	Aniline-2.5-disulphonic acid	1-2	5	Similar to Pontacyl Light Yellow 3 G, but duller	
23	Cleve's acid	2	5	Similar to du Pont Orange G, but considerably duller	
24	1-Naphthylamine-4.8-disulfonic acid	2	5 G	Near to Metanil Yellow 1955	
25	2-Naphthylamine-6.8-disulfonic acid	2-3	4	Similar to du Pont Azo Yellow conc., but brighter and yellower	1/2 Strength
26	H acid	3	5	Little like National Croceine Orange Y conc., but very much duller	

Monoazo Dyes Made With Citrazinic Acid

Dyed as Chrome Top Colors

No. of Dye	Diazo Component	Light Fastness	Washing Fastness	Shade	Strength
1C.	Aniline		5	Similar to Pontachrome Flavine A	1/2 Strength
2C.	∝-Naphthylamine			Similar to Pontachrome Yellow 3R N, but yellower	Weaker
3C.	o-Anisidine			Yellow; different from others, this being brighter and yellower	
4C.	p-Amino-acetanilide			Close to Pontachrome Yellow GR, but little yellower	
5C.	2.5-Dichloraniline	2-3	5	Similar to Pontachrome Yellow 6G, but little brighter and yellower	1/2 Strength
6C.	p-Nitraniline	3	3 R	Very close to Pontachrome Fast Yellow R	
7C.	Picramic acid	2	2	Somewhat like Pontachrome Red A4B, but much bluer	1 1/2 Strength
8C.	Anthranilic acid	2	2-3 R	Similar to Pontachrome Yellow 3 R N, but brighter and yellower	Much Weaker
9C.	p-Aminobenzoic acid	2-3	2	Similar to Pontachrome Yellow GS, but brighter and yellower	Much weaker
10C.	Sulfanilic acid	2	3 R		
11C.	Metanilic acid			Near to Pontachrome Flavine A, but brighter	Little weaker
12C.	Naphthionic acid	2	2	Similar to Pontachrome Yellow 3 R M, but much yellower	Much weaker
13C.	2-Naphthylamine-1-sulfonic acid	2-3	3	Yellow	
14C.	2-Naphthylamine-6-sulfonic acid	2-3	2	Yellow; nothing else as bright	
15C.	2-Naphthylamine-7-sulfonic acid	2-3	2	Similar to Pontachrome Yellow G R, but brighter and yellower	
16C.	1-Naphthylamine-5-sulfonic acid	2	3	Similar to Pontachrome Yellow 3 R M, but brighter and yellower	Much weaker
17C.	1-Naphthylamine-8-sulfonic acid	3	4	Similar to Pontachrome Yellow G R, but brighter and yellower	
18C.	p-Nitraniline-o-sulfonic acid			Near to Pontachrome Flavine A, but little yellower	Little weaker
19C.	o-Aminophenol-p-sulfonic acid	1-2	2	Somewhat like Pontachrome Red A4B, but yellower	
20C.	1-Amino-2-naphthol-4-sulfonic acid	2-3	3	Similar to du Pont Alizarine Red S, but much bluer	Bluer
21C.	Aminoazobenzene-p-sulfonic acid	2	2	Similar to Pontachrome Yellow 3 R N, but brighter and yellower	1/2 Strength
22C.	Aniline-2.5-disulfonic acid			Similar to Pontachrome Yellow G R, but little brighter and little yellower	1/2 Strength
23C.	Cleve's acid	2	3	Similar to Pontachrome Yellow 3 R N, but brighter and yellower	1/2 Strength
24C.	1-Naphthylamine-4.8-disulfonic acid	2	4 G	Bright orange	
25C.	2-Naphthylamine-6.8-disulfonic acid	2-3	2	Similar to Pontachrome Yellow G R, but much brighter and yellower	3/4 Strength
26C.	H acid	3	3 B	Similar to Pontachrome Red A4B, but duller and bluer	3/4 Strength

colors showed, as a rule, considerable improvement, those of the diazo dyes being much better. The color that bleeds off from some of the dyed wool does not have the property of staining either white worsted, silk, cotton, rayon or acetate silk. This is true, f. i. of dyes prepared from citrazinic acid with anthranilic acid (No. 8), p-aminobenzoic acid (No. 9), 2-naphthylamine-1-sulfonic acid (No. 13), sulfanilic acid (No. 10), etc. Some of the dyes, such as the monoazo dyes made from citrazinic acid with ∝-naphthylamine (No. 2), p-nitraniline (No. 6), and the disazo dyes generally dye all the fibers. Washing changes the shades of some of the dyes as they are evidently very sensitive to alkali.

This sensitivity to washing may possibly be obviated

Disazo Dyes Made With Citrazinic Acid

Dyed as Acid Colors

No. of Dye	Diazo Components	Light Fastness	Washing Fastness	Shade	Strength
27.	2 Mols. p-Nitraniline	3		Rust color;different from du Pont or National colors	
28.	2 Mols. Sulfanilic acid		5	Dull brown;different from du Pont or National colors	
29.	2 Mols. Naphthionic acid	2-3	5 R	Nearest to National Resorcin Brown R M	
30.	2 Mols. o-Aminophenol-p-sulfonic acid	3	5 R	Brown;different from du Pont or National colors	
31.	Sulfanilic acid+p-Nitraniline	3		Brown;different from du Pont or National colors	
32.	Sulfanilic acid+1-Amino-2-Naphthol-4-sulfonic acid	3	4	Nearest to du Pont Naphthylamine Black V	1/2 Strength
33.	Benzidine(+2 Mols.Citrazinic acid)	3		Crimson;different from du Pont or National colors	

Dyed as Chrome Top Colors

27C.	2 Mols. p-Nitraniline	2-3		Nearest to Pontachrome Yellow 3 R N	Very weak
28C.	2 Mols. Sulfanilic acid		2 R	Nearest to Pontachrome Yellow 3 R N,but brighter	
29C.	2 Mols. Naphthionic acid	2	1	Nearest to Pontachrome Yellow R G, but redder	Weaker
30C.	2 Mols. o-Aminophenol-p-sulfonic acid	1-2	2	Similar to Pontachrome Red A4B, but little brighter (and yellower	
31C	Sulfanilic acid+p-Nitraniline	3		Nearest to Pontachrome Yellow 3 R N	1/2 Strength
32C.	Sulfanilic acid+1-Amino-2-naphthol-4-sulfonic acid	2-3	1	Reddish dull black: poor dye	
33C.	Benzidine(+2 Mols.Citrazinic acid)	3		Nearest to Pontachrome Red A4B	

Monoazo Dyes Made With Citrazinic Acid Mono Butyl Ether

Dyed as Acid Colors

No. of Dye	Diazo Component	Light Fastness	Washing Fastness	Shade	Strength
34.	Anthranilic acid	1-2	4	Practically identical with Pontacyl Light Yellow 2 G	1 1/2 Strength
35.	Sulfanilic acid	3	4	Similar to National Metanil Yellow 1955, but duller (and redder	
36.	Naphthionic acid	2	2	Similar to du Pont Orange II Conc., but duller	1/2 Strength
37.	2-Naphthylamine-6-Sulfonic acid	3	3	Red; different from du Pont or National colors	
38.	Aminoazobenzene-p-sulfonic acid	2	3 R	Similar to du Pont Orange II conc.,but much duller (and redder	
39.	Aniline-2.5-disulfonic acid	2	5	Almost identical with Quinoline Yellow	Stronger
40.	2-Naphthylamine-6.8-disulfonic acid	3	2	Very close to du Pont Metanil Yellow	1/2 Strength

Dyed as Chrome Top Colors

34C.	Anthranilic acid	2-3	3 R	Similar to Pontachrome Yellow 3 R N, but yellower	1/2 Strength
35C.	Sulfanilic acid	3	3 R	Similar to Pontachrome Yellow S W, but little (brighter	
36C.	Naphthionic acid	3	2	Similar to Pontachrome Yellow 3 R N, but little (yellower	1/2 Strength
37C.	2-Naphthylamine-6-sulfonic acid	3	2	Similar to Pontachrome Brown G	1 1/4 Strength
38C.	Aminoazobenzene-p-sulfonic acid	2	3 R	Similar to Pontachrome Yellow 3 R N, but little (brighter and redder	1/2 Strength
39C.	Aniline-2.5-disulfonic acid	2	4	Very similar to Pontachrome Yellow 6 G, but (brighter	Much weaker
40C.	2-Naphthylamine-6.8-disulfonic acid	3	2	Very similar to Pontachrome Yellow G R	Weaker

No. of Dye	Citrazinic Component	Diazo Component		Class of Dye	Shade
41.	Citrazinic acid	p-Toluidine		Monoazo	Yellow
42.	" "	Dehydrotoluidine		"	Golden Yellow
43.	Citrazinic acid methyl ester	Aniline		"	Yellow
44.	" " " "	p-Toluidine		"	Very bright yellow
45.	" " ethyl ester	Aniline		"	Yellow
46.	" " " "	Naphthionic acid		"	Orange
47.	" " mono butyl ether	Aniline		"	Golden yellow
48.	" " " " "	p-Toluidine		"	Yellow
49.	" " " " "	Dehydrotoluidine		"	Brownish yellow
50.	" "	p-Toluidine	2 Mols.	Disazo	Red
51.	" "	Dehydrotoluidine	"	"	Brown
52.	" " methyl ester	p-Toluidine	"	"	Reddish brown
53.	" " " "	p-Nitraniline	"	"	Brownish orange
54.	" " " "	Naphthionic acid	"	"	Brownish red
55.	" " " " (2 Mols.)	Benzidine		"	Reddish Brown
56.	Citrazinic acid ethyl ester	Aniline	2 Mols.	"	Yellowish brown
57.	" " " "	Naphthionic acid	"	"	Purple brown
58.	" " mono butyl ether	Aniline	"	"	Red
59.	" " " " "	p-Toluidine	"	"	Red
60.	" " " " "	Dehydrotoluidine	"	"	Orange brown
61.	" " " " "	Naphthionic acid	"	"	Brown
62.	" " " " " (2 Mols.)	Benzidine		"	Purple brown

(Concluded on Page 524)

Effective
MILDEW-RESISTANT TREATMENTS
for Cotton Fabrics

MARGARET S. FURRY and HELEN M. ROBINSON
Assistant Textile Chemist *Formerly Assistant Scientific Aide*

Textiles and Clothing Division, Bureau of Home Economics,
United States Department of Agriculture[1]

IF properly chosen, mildew-resistant finishes will markedly prolong the life of such house furnishings as covers for porch furniture and shower curtains as well as sails, tarpaulins, and other canvas equipment. To be completely satisfactory, however, these finishes should have other characteristics. They should be comparatively easy to apply; they should not decrease the strength of fabrics or cause excessive shrinkage; they should withstand weathering and repeated laundering and they should be colorless, odorless, and non-toxic to human beings.

The United States Bureau of Home Economics has therefore extended a recent study of such finishes[2]. Various treatments have been investigated to determine the effect they have on the breaking strength of the fabric and to obtain more conclusive data on their mildew-preventing power. Information on the effectiveness of mildew-resistant treatments was obtained in terms of the change in breaking strength of the treated fabrics after inoculation and incubation with a micro-organism.

An 8-ounce unbleached cotton duck was used. It was degreased and desized before the special finishes were applied. Because of the non-uniformity of the fabric (the variation in warp breaking strength was 9 per cent[3]) a random method of sampling the fabric for the various chemical treatments was used.

The fabric to be treated was divided first into blocks of approximately two yards each. This was cut into strips for the raveled-strip breaking-strength test in order to have two paired samples for each of the different chemical treatments and for three untreated controls. The strips for the treatments and controls were chosen at random. One set of the paired strips was used in obtaining the change in breaking strength of the treated fabric due to the chemical treatment alone and the other set for determining the change in strength due to the combined effect of treatment and mildew growth. Ten blocks of fabric were used in the experiment since it seemed necessary to have at least 10 replicates for each treatment.

In order to bring the fabric strips to the same moisture content before conditioning, they were dried in an electric oven at 35°-40° C. for 18 hours. All samples were conditioned in a constant temperature and humidity room maintained at 70° F. and 65 per cent relative humidity for 24 hours and then were weighed.

The details of the treatments, some of which required immersing the fabric in more than one chemical solution, are given in Table 1. Each strip of fabric was processed separately in 50 ml. of the finishing bath for the stated temperature and time. The strip was then quickly removed, placed between two sheets of blotting paper, and a roller weighing one kilogram was drawn over the blotting paper. When dry, the fabric strip was placed in an oven maintained at 35°-40° C. for 18 hours, and again conditioned and weighed. The percentage gain in weight due to the finishing treatment was calculated.

The mildew resistance of the treated strips of cotton duck was determined by measuring their change in breaking strength after inoculation and incubation with *Chaetomium globosum*, Kunze[2]. According to the routine bacteriological procedure most of the strips were steam sterilized. In a few cases, however, this process was omitted since in the previous study it was noticed that steaming affected the finishing treatments that had been applied. For those that were inoculated without sterilization, the same care and sterile technique were used in handling them. All samples were examined for sterility before inoculation and for amount of growth on the third, seventh, and fourteenth day of incubation.

At the end of the 14 days, the strips were removed from the bottles in which they were incubated and carefully rinsed in running water to remove the mildew growth. After drying in air they were again placed in the oven at 35°-40° C. for 18 hours. The samples were conditioned for at least 12 hours in the humidity room and breaking strength measurements made on the strips, using the motor-driven Scott tester.

Breaking strength tests were also made on similarly conditioned, sterilized but not inoculated strips of the

(Continued on Page 520)

[1]Acknowledgment is made to Harry Humfeld, formerly associate bacteriologist, Division of Cotton and Other Fiber Crops and Diseases, Bureau of Plant Industry, for suggestions concerning the study and to Dorothea Klemme, junior chemist, Textiles and Clothing Division, Bureau of Home Economics, for assistance with the microbiological work.

[2]Mildew-resistant treatments on fabrics by Furry, Robinson and Humfeld. Industrial and Engineering Chemistry 33 (No. 4): 538-545, 1941.

[3]The coefficient of variation is the standard deviation expressed as a percentage of the mean.

PROCEEDINGS OF THE

American Association of Textile Chemists and Colorists

SEPTEMBER 29, 1941

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Members should notify the **Secretary** immediately of all address changes

REPORT OF THE NOMINATING COMMITTEE

THE Councilors Representing Sections, in their capacity as Nominating Committee, have directed the Secretary to report the following nominations for the election of 1941:

For President—Thomas R. Smith.

For Vice-Presidents—Roland E. Derby and Charles A. Seibert.

Under the Constitution, additional nominations, each signed by at least twenty-five Senior members, filed with the Secretary not later than October eighth, will be given equal standing on the ballot.

Respectfully submitted,

HAROLD C. CHAPIN, *Secretary.*

TECHNICAL PROGRAM

Annual Meeting and Convention
October 31st–November 1st
The Carolina, Pinehurst, N. C.

Kenneth H. Barnard
Chairman, Technical Program Committee

Mr. Barnard is chief chemist of the Print Works Division of the Pacific Mills in Lawrence, Mass. and has been an active member of the Research Committee for many years. He has arranged an interesting, varied and timely program of technical papers.

JUDGES ANNOUNCED FOR INTERSECTIONAL CONTEST

KENNETH H. BARNARD, chairman of the technical program committee for the annual meeting, has announced the judges for the intersectional contest to be held Friday afternoon, October 31st. The judges and the sections they represent are as follows:

Northern New England—ROLAND E. DERBY, president of the Derby Co. and vice-president of the A.A.T.C.C.; *Rhode Island*—A. E. HIRST, sales manager, Standard Chemical Co. and charter member of the A.A.T.C.C.; *New York*—HENRY F. HERRMANN, sales administrator, General Dyestuff Corp. and the "Daddy" of the Intersectional Contest; *Philadelphia*—DR. GLEN HIERS, chief chemist, Collins & Aikman Corp.; *Piedmont*—HERMAN WALKER, superintendent, Morganton Full Fashioned Hosiery Co.; *Southeastern*—GEORGE E. MISSBACH, sales representative, F. A. Ross & Co., Inc.; *South Central*—WALTER E. HADLEY, chief chemist, Standard Coosa Thatcher Co. and charter member and past secretary of the A.A.T.C.C.; *Mid-West*—ARTHUR T. BRAINERD, manager, Chicago office, Ciba Co., Inc.

The "chief justice" will again be DR. LOUIS A. OLNEY, head of the department of chemistry and dyeing, Lowell Textile Institute, and president emeritus and chairman of the research committee of the A.A.T.C.C.

The above-named judges, who have been active in local and national affairs of the association, are all qualified to expertly judge the various papers which have been entered in the contest. We are assured that the decisions which they render will be fair and impartial.

TEXTILES AND NATIONAL DEFENSE

ONE of the features at the annual meeting technical program on Saturday morning, November 1st, will be a session devoted to "Textiles and National Defense." This will be presided over by Dr. Carl Z. Draves, president of the A.A.T.C.C. The entire program has been thoroughly coordinated to give a complete, up-to-the-minute picture of the textile industry at this time of national emergency, by men who know their subject and are well qualified to express it. The importance and timeliness of this session can readily be appreciated by all members. The subjects to be discussed and the speakers are as follows:

"1941 in the Dyestuff Industry"—By ALBERT E. SAMPSON, National Aniline & Chemical Co., Inc.—a man well qualified by wide experience to present the outlook of the dye manufacturers.

"The Chemical Manufacturer and the National Emergency"—by Dr. D. H. POWERS, Rohm & Haas Co., Inc.—an able and well known speaker who will discuss a subject nearest his heart.

"Defense Problems from the Mill Man's Viewpoint"—by WINN W. CHASE, associate editor, *Textile World*, whose articles and speeches deserve well merited attention due to his bird's eye view of the textile mills.

"Purchase and Inspection of Textiles for the Army"—by LIEUT. COLONEL VERE PAINTER, representing the Philadelphia Quartermaster Corps, War Department—one of the men whose job has not merely doubled or tripled, but increased twenty-five fold and who will tell how he handles it.

"Textiles from the Navy Standpoint"—by LIEUT. R. G. BUCK, S.C., U.S.N., Brooklyn Naval Clothing Depot. His story, like Colonel Painter's, will describe what our armed forces expect and are obtaining from the textile industry.

"Production of Wools of Unusual Properties through Modification of their Chemical Structures"—by DR. MILTON HARRIS, Textile Foundation, National Bureau of Standards, Washington, D. C.,—report on a brilliant piece of research work with far reaching results to the users of wool.

TECHNICAL PROGRAM ON HOSIERY

PARTICULAR interest is being focussed on the hosiery industry at the present time due to the cessation of raw silk imports from Japan. Accordingly the technical program committee has arranged a series of coordinated papers on the hosiery industry to be presented at the Saturday morning technical session at the annual meet-

ing. This session is bound to have great popularity as well as technical interest, particularly as Pinehurst is near the center of the hosiery industry in the south. The program will be under the direction of T. R. Smith, Wiscassett Mills Co. and vice-president of the A.A.T.C.C. It will consist of the following papers:

"The Dyeing of Army Socks"—by S. M. HAMILTON, Holeproof Hosiery Co.

"The Throwster Serves the Silk Hosiery Industry Without Silk"—by GEORGE URLAUB, textile consultant.

"The Dyeing of Nylon Hosiery"—by P. H. STOTT, technical laboratory, E. I. Du Pont de Nemours & Co., Inc.

"The Manufacturing of Full Fashioned Hosiery in Relation to the Finish"—by JOSEPH KELLY, May Hosiery Mills.

"The Dyeing and Finishing of Full Fashioned Hosiery"—by VIRGIL T. HARTQUIST, Wayne Knitting Mills.

The speakers are all well-known in the hosiery industry and are capable of presenting an authoritative discussion of a subject which is so much in the public eye today.

CLINIC ON LIGHT FADING

FOLLOWING the sessions on national defense and hosiery at the Saturday morning technical program of the annual meeting there will be a "clinic" on light fading. In this session there will be representatives of the manufacturers of the two leading fading lamps speaking as well as a consumer who has done a lot of work on the variables encountered in using fading lamps. This "clinic" will be of special interest to everyone interested in light fading and it is hoped that many constructive suggestions will result from it. Following is the program of papers:

"Fundamental Concepts in Fade-Ometer Design and Operation"—by E. H. HARVEY, Atlas Electric Devices Co.

"The 'National' Accelerated Fading Unit"—by C. G. OLLINGER, National Carbon Co., Inc.

"Action of Accelerated Fade Tests on the Color of Wool Fabrics"—by J. J. GLENN, Sidney Blumenthal Co., Inc.

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TRANSPORTATION SCHEDULES
To Pinehurst, N. C. for Annual Meeting

REVISED AND LAST MINUTE CONDENSED SCHEDULES FOR MEMBERS ARRIVING FROM BOSTON, PROVIDENCE, NEW YORK, PHILADELPHIA AND WASHINGTON, D. C.

Railway-Air Lines and bus company officials suggest that railway passengers use the following south-bound schedule:

Railway Schedule

Leave Boston	11:00 A.M.
Leave Providence	12:05 P.M.
Leave New York	6:30 P.M.
Leave Philadelphia	8:10 P.M.
Leave Washington	11:45 P.M.
Arrive Pinehurst	8:35 A.M.

Returning, north-bound by railway, there are three train connections to be considered.

Leave Southern Pines	11:10 P.M.
Arrive Washington, D. C.	8:25 A.M.

with connections to

Arrive Philadelphia	11:12 A.M.
Arrive New York	12:50 P.M.
Arrive Providence	4:55 P.M.
Arrive Boston	5:55 P.M.

Those desiring to make this connection would be obliged to leave the banquet before all ceremonies have been concluded.

Alternative Schedule—North-Bound

Leave Southern Pines	4:18 A.M.
Arrive Washington	11:45 A.M.
Arrive Philadelphia	2:31 P.M.
Arrive New York	4:15 P.M.
Arrive Providence	7:55 P.M.
Arrive Boston	8:55 P.M.

Buses will be available for those north-bound passengers desiring to make connections at Southern Pines at 4:18 A.M. No sleeping cars can be set aside at Southern Pines or Pinehurst for this connection. However, if there are sufficient passengers interested in sleeping-car accommodations, sleeping cars will be set aside at Hamlet, N. C., approximately 30 miles from Pinehurst, which will be available after 11:00 P.M. These cars will be picked up and attached to the train leaving Hamlet, N. C., 3:45 A.M. (This is the same train that stops at Southern Pines at 4:18 A.M.)

RESERVATIONS FOR SLEEPERS TO BE SET ASIDE AT HAMLET MUST BE MADE NOT LATER THAN 1:00 P.M. FRIDAY, OCTOBER 31. SUCH RESERVATIONS SHOULD BE MADE WITH THE TRANSPORTATION PORTER OR THE INFORMATION COMMITTEE CHAIRMAN, ROBERT A. BRUCE AT THE CAROLINA HOTEL, FRIDAY MORNING.

Third Alternative Schedule

For those desiring to stay over Sunday to enjoy the delightful climate and sports at Pinehurst, there is a *train*

Leaving Pinehurst	6:30 P.M.
Arriving Washington	3.55 A.M.

(sleeping cars on this train parked for occupancy until 8:00 A.M.)

Arrive Philadelphia	7:13 A.M.
Arrive New York	8:55 A.M.
Arrive Providence	1:55 P.M.
Arrive Boston	2:55 P.M.

Bus Schedule Between Raleigh and Pinehurst, N. C.

For members planning to use Air Lines to and from Raleigh, we suggest the following *bus* schedules from Raleigh to Pinehurst and return:

Leave Raleigh	8:00 A.M.	or	2:30 P.M.
Arrive Pinehurst	11:00 A.M.		4:30 P.M.

Returning:

Leave Pinehurst	10:25 A.M.
Arrive Raleigh	12:40 P.M.

There is only one bus schedule north-bound.

This connection will enable north-bound air passengers to connect with the plane leaving Raleigh, Eastern Airline Plane, trip no. 14.

Leave Raleigh	3:05 P.M.
Arrive New York	7:15 P.M.

connecting with American Airlines, trip no. 20.

Leave New York	7:30 P.M.
Arrive Boston	8:45 P.M.

It is urgently suggested that you make your Pullman reservations for your return trip at the same time you purchase your transportation to the convention.

MEETING, SOUTHEASTERN SECTION

AT its regular meeting, held on Saturday evening, September 6th, at the Hotel Dempsey, Macon, Ga., the Southeastern Section of A.A.T.C.C. devoted the entire meeting to an informal, spontaneous round-table discussion of a set of practical plant problems. There were no prepared speeches or addresses, the whole evening being devoted to impromptu, unrehearsed and general discussion by those present of several topics relating to dyeing, bleaching and finishing.

The questions had been developed by the program committee based on suggestions submitted in advance by the individual members.

Perrin N. Collier, Vice-President and Director of Research, Callaway Mills, LaGrange, Ga., presided as chairman of the section. The discussion was in charge of Robert J. McCamy, Superintendent of Dyeing, Pepperell Manufacturing Company, Lindale, Ga., who was chairman of the program committee, and who handled the discussion of the questions on dyeing. John P. Harrison, Superintendent, Muscogee Manufacturing Co., Columbus, Ga., led the dismussion on bleaching subjects, and Leslie L. Bamberger, Purchasing Agent, Lanett Bleachery and Dye Works, conducted the discussion on the subjects relating to finishing.

There was splendid response from the members of the audience in discussing the questions and it was unanimously voted to conduct another meeting of this type in the near future. The next meeting of the section will be held in Atlanta, Ga., on December 6th.

A copy of the questions discussed at the Macon meeting follows:

* * *

Questionnaire for Round-Table Discussion

Meeting of Southeastern Section, A.A.T.C.C.
Hotel Dempsey, Macon, Ga.
Saturday, September 6, 1941

Dyeing

1. What is the most effective way of controlling shades from batch to batch, and the most effective method of reworking batches up to shade, especially vat-dyed colors?

2. What are the relative advantages and disadvantages of the pigment method of dyeing vat colors as compared with the reduced method?

3. (a) What are your major problems in beam and package dyeing? How do you secure desired density and get good work with a minimum of waste? (b) Why is yarn dyed in wound form with vat colors always lighter on the outside of the package than it is in the center or down next to the core on which it is dyed? (c) What are the causes and remedies of sulfur black tendering?

4. Is there a way to soften heavy sulfur shades that have become harsh in dyeing?

Robert W. Philip, Editor of "Cotton" and councilor of the Southeastern Section, made this snapshot for the American Dyestuff Reporter during the meeting of the Southeastern Section at Macon, Ga., on September 6. It shows, left to right: John P. Harrison, superintendent, Muscogee Mfg. Co., Columbus, Ga., who led the discussion on bleaching; (standing) Robert J. McCamy, superintendent of dyeing, Pepperell Mfg. Co., Lindale, Ga., chairman of the program committee who conducted the discussion on dyeing; Perrin N. Collier, vice-president and director of research, Callaway Mills, LaGrange, Ga., chairman of the section; and Walter M. Scott, chief of the cotton chemical finishing division of the Southern Regional Research Laboratory, New Orleans, La., now a member of Southeastern section.

Bleaching

5. What are the advantages and disadvantages of chlorine bleaching versus peroxide bleaching?

Finishing

6. (a) What is the most effective method for controlling variations in shrinkage in the Sanforizing process? (b) Please discuss the care and most efficient use of Sanforizing blankets.

7. What are the advantages and disadvantages of the cation active finishing agents?

Robert J. McCamy, Chairman,
William B. Griffin,
George E. Missbach,
PROGRAM COMMITTEE

* * *

Respectfully submitted,

CURT MUELLER,
Secretary.

◆ ◆

OUTING, NORTHERN NEW ENGLAND SECTION

UNDETERRED by war clouds or threatening priorities, two hundred and fourteen members and guests of the Northern New England Section journeyed on June 6, 1941 to the Vesper Country Club, Lowell, Mass., for the Nineteenth Annual Outing of the Section.

It was a perfect June day, neither too hot nor too cool. Eager claimants thronged the golf course, played tennis, pitched horseshoes, or vociferously played at baseball. Others less athletically inclined sat around or strolled about in groups conversing and watching contestants.

The last putt having been sunk, tennis ball served, horseshoe pitched, the baseballers having ceased their toil and ethical comments, and salesmen having successfully

avoided taking orders, happy and hungry, the crowd trooped to the main building at Vesper for dinner.

Dinner, varied to suit different tastes, was satisfying and efficiently served. Thanks are due to George Stewart and members of the Dinner Committee and to the steward and personnel of the Club. Would-be virtuosi and aspiring choruses interspersed eating with the rendering (rending) of favorite ditties and popular airs.

John Dalton, Chairman of the Section, called together the post-dinner meeting and in a few well chosen words welcomed members and guests. He announced that Dr. Olney for the first time in many years was absent from the Outing and was attending at Lehigh University the 45th reunion of his class. He turned the meeting over to Arthur Sampson, the Chairman of the Outing Committee.

"Art" praised the Section for many accomplishments, among which may be mentioned turning out in such record numbers, success in praying for a fair day, and especially the remarkable achievement of sending in cards with dinner reservations. May it become a habit! He then introduced George Linberg, Chairman of the Golf and Prize Committees.

George proclaimed, and in spite of rising and tumultuous applause, made it understood that he proposed to carry out the distribution of prizes *"expeditiously"* by a new plan, viz, to call the names of winners for an event all together instead of individually.

He first announced the award to Arthur Sampson, Chairman of the Outing Committee, of an unusually valuable and rare prize, viz,—a six ounce vial of Formaldehyde.

He then proceeded with the expeditious distribution of prizes by the new plan and in very short order this pleasing part of the program was completed with great general satisfaction. Following is the list of the lucky and successful contestants.

Golf—Members

Class A

1st low gross.....M. P. Graham (80)
2nd low gross.....H. Ross
3rd low gross.....M. H. Mettee
1st low net.....Fred Dushame
2nd low net.....S. Sheppard
3rd low net.....J. M. Sorbin

Class B

1st low gross.....W. A. Dainton
2nd low gross.....John Dalton
3rd low gross.....A. G. Hibarger
1st low net.....C. E. Fickinger
2nd low net.....J. Cummings
3rd low net.....W. C. McElroy

Class C

1st low gross.....S. C. Clement
2nd low gross.....E. S. Chapin
3rd low gross.....W. A. Kingman
1st low net.....Neil Buckley
2nd low net.....H. F. Clapham
3rd low net.....Geo. A. Stevens

Golf—Non-Members

Class A

1st low gross.....J. Mullaney (78)
2nd low gross.....W. P. Fuller
3rd low gross.....H. C. Milton
1st low net.....F. L. Sjostrom
2nd low net.....K. Batchelor
3rd low net.....M. Flynn

Class B

1st low gross.....A. W. Sulloway
2nd low gross.....R. L. Brown
3rd low gross.....V. Tessier
1st low net.....F. Roache
2nd low net.....F. Wilson
3rd low net.....M. Shaw

High Gross.....W. H. Alexander (200)
Nearest to pin at 14th.....A. H. Pierce

Al Pierce and Phil Graham had their usual close contest. Phil's ball was 3 feet from the pin, Al's 3 feet six inches, but Phil already had low gross. Three weeks later at the Drysalters' Outing Al zoomed across with low net (78) with Phil second (81). Such golf!!!

Putting—Members

1st .....M. Lane
2nd .....D. Gifford

Putting—Non-Members

1st .....H. Wieland, Jr.
2nd .....A. Mullin

Tennis

Singles

1st .....Morris Lewis
2nd .....K. Barnard

Doubles

A. L. Wilson — Gene Bernard

Horseshoes

Singles

1st .....A. C. Dege
2nd .....James Monk

Doubles

M. W. Hardie — G. L. Boehm

Ringer after ringer! How do you do it fellows?

Baseball

W. B. Luce and his Yankees

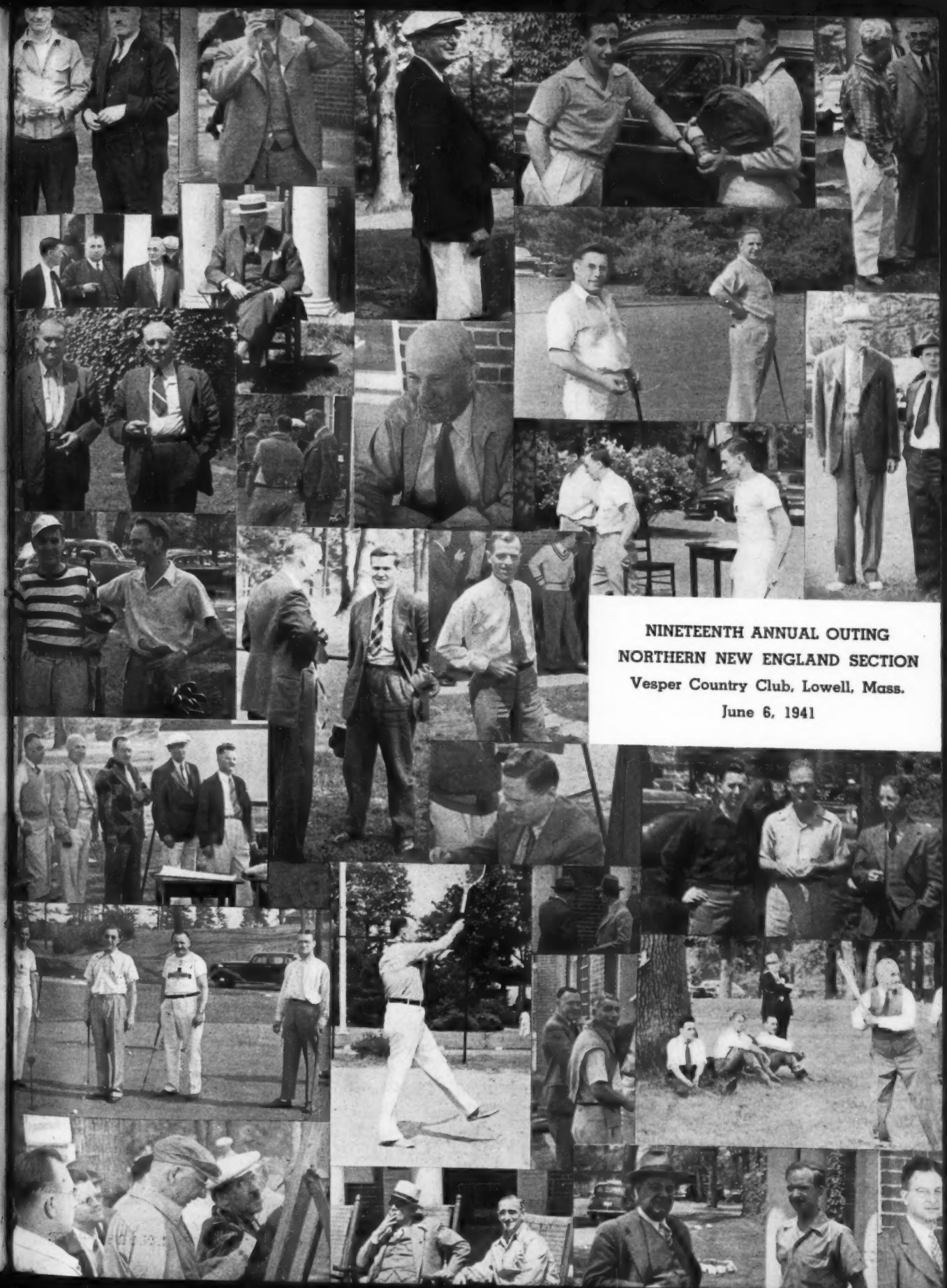
Joe Travers and his Red Sox failed to come through this year even with the help of Umpire Finlay.

The thanks of the Section are due to the following Outing Committee: *Chairman*—Arthur H. Sampson; *Chairman Ex Officio*—John N. Dalton; *Committee for Golf*—George O. Linberg, Earl R. Rutter, D. Scott Clement and Edward Bell; *Committee for Tennis*—Kenneth H. Barnard; *Committee for Horseshoes*—Frank J. Robertson; *Committee for Soft Ball*—Joseph E. Travers and Harry F. Finlay; *Committee for Dinner*—George Stewart, Frank J. Rizzo, Delbert E. Ray and Henry T. Rooney; *Committee for Program*—Edward S. Chapin.

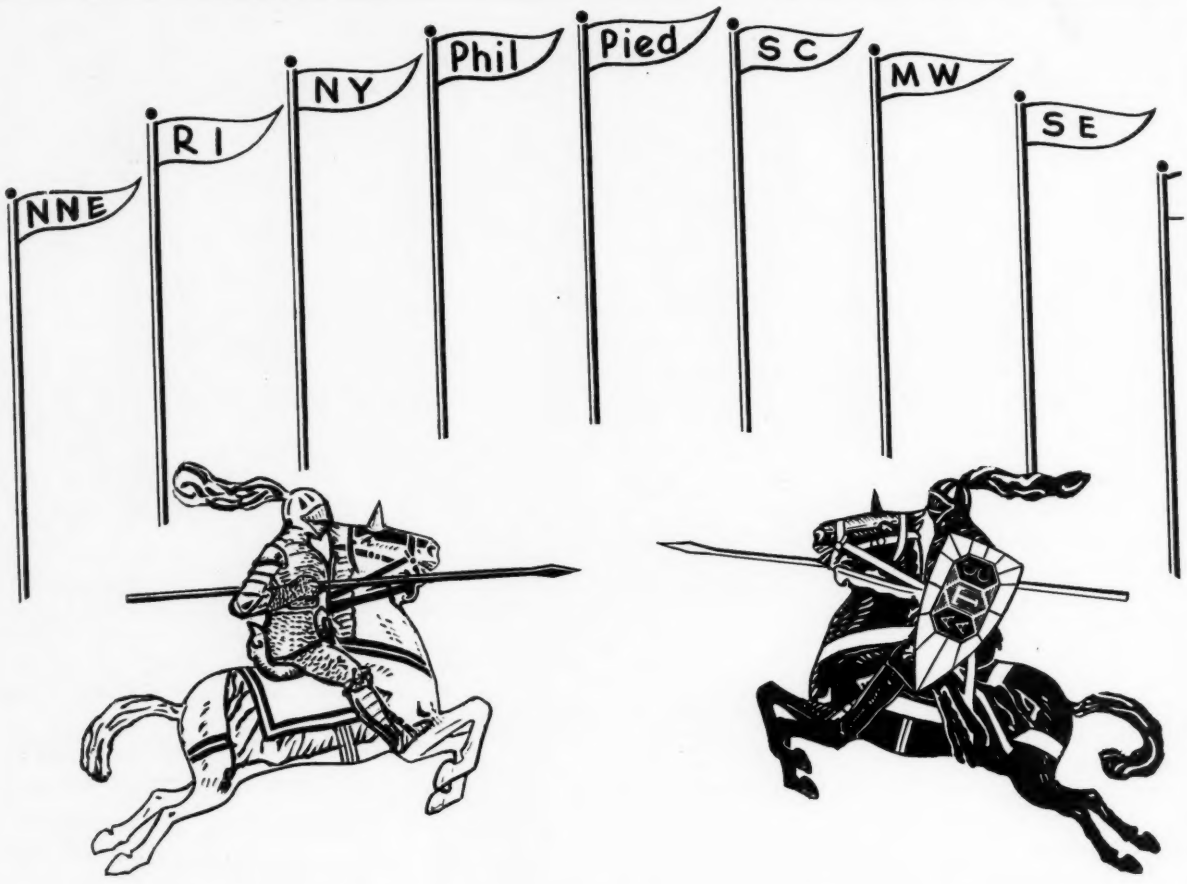
Acknowledgment should also be made to numerous firms and persons, who generously donated prizes, to the DYESTUFF REPORTER, to Mr. Johnson, managing editor of the DYESTUFF REPORTER and Dr. Harold Chapin, National Secretary, who jointly took the pictures shown on an adjoining page.

Respectfully submitted,

EDWARD S. CHAPIN, *Secretary*



NINETEENTH ANNUAL OUTING
NORTHERN NEW ENGLAND SECTION
Vesper Country Club, Lowell, Mass.
June 6, 1941



Intersectional — Contest

Titles of papers to be entered by the various sections

Northern New England—"The Prediction and Control of Colored Blends by Optical Means."

Rhode Island—"Textile Chemists Meet National Defense Emergency."

New York—"Cotton Fabric Constructions in Relation to Water Resistance."

Philadelphia—"Testing Vat Dyed Cotton for Fastness to Hypochlorite Bleach."

Piedmont—"Relative Exhaustion of Vat Colors in the Pad Pigment Method of Jig Dyeing."

Southeastern—"Progress Report on the Preparation and Processing of Domestic Flax."

South Central—"Evaluation of Finishing Agents for Sanforizing."

The names of the judges appear on page P506

Annual Meeting and Convention
October 31st—November 1st, 1941

ELECTROCOATING FABRICS*

J. O. AMSTUZ

Chief Engineer, Behr-Manning Corp.

THE underlying principle of electrocoating fabrics has been known to mankind for more than 2,500 years. It is demonstrated whenever a high school teacher rubs a glass rod with a piece of silk, causing bits of paper to stand on end before jumping to the rod and sticking there in an upright position. We say that the glass rods and the bits of paper are electrified or charged. We know now that most substances possess this property to some extent. By experimenting with many substances it was found that some repelled each other and some attracted each other. It was concluded that two kinds of electricity existed which were defined arbitrarily as positive and negative. The first law applied in electrocoating emerged and took useful form in Benjamin Franklin's time. It stated that equally charged substances repel each other while unequally charged substances attract each other. This fundamental law of electrostatics is used in electrocoating fabrics, and it was first applied in the coating industry for the manufacturing of sandpaper when in 1926 Mr. E. C. Schacht of the Behr-Manning Corporation, made his first experimental run. The process was adapted in 1939 to the making of dress goods and other fabrics. Electrocoating fabrics presents many interesting problems to the textile chemist and colorist. The problems may be grouped as follows: (1) electrocoating; (2) fiber preparation; (3) adhesives.

ELECTROCOATING

The equipment for electrocoating is relatively simple. Small crushed mineral particles, such as flint, garnet, aluminum oxide, silicon carbide, emery, or fibers of rayon, wool, silk and cotton, are carried by a belt conveyor into a strong electrostatic field. Moving over the belt conveyor is a ribbon or web of adhesively coated paper or cloth, calling the backing. Two electrodes, similar to condenser plates, are required to establish the electrostatic field. One of the electrodes is located underneath the conveyor belt. The other one is placed above the backing. The arrangement is shown in Fig. 1.

The top electrode is grounded and the bottom electrode is connected to the high tension terminal of a transformer or a kenotron rectifier set. Electrode spacing and voltage between the electrodes are adjustable. The voltage is varied from 20,000 to 70,000 volts while the electrode spacing is held from ½" to 1½".

*Presented at meeting, New York Section, May 23, 1941.

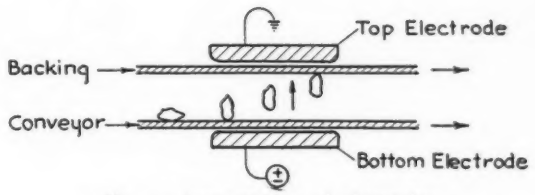


Fig. 1—Arrangement of electrodes.

A commercial coating machine does require only minor changes in order to be transformed into an electrocoater. The adhesive application, the curing and the takedown equipment remain unchanged. The only necessary alterations are the rearrangement of the fiber feed and the addition of an electrocoating unit. Figure 2 illustrates how this is accomplished. The operation of the coating unit is similar to any coating machine.

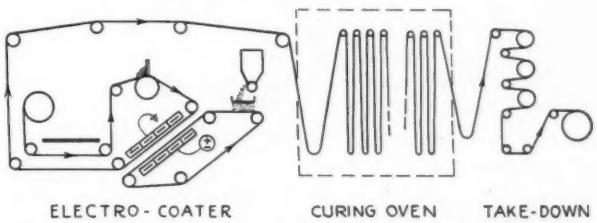


Fig. 2—Diagrammatic sketch of an electrostatic coating unit.

The backing, paper or cloth, is coated on one side with a suitable adhesive. This can be done by means of a doctor blade as shown in Fig 2. or with a pair of coating rolls. The coated backing, with the adhesive facing down, is passed in between the electrodes. Finely divided particles are fed uniformly from a hopper through a screen onto a conveyor belt which carries the particles into the electrode spacing. As soon as the particles enter the electrode spacing they are polarized and charged due to the high intensity field which exists between the electrodes, and since the lower electrode is connected to the high side of a potential source, particles in contact with the lower electrode acquire a charge which is of the same polarity as the bottom electrode.

The particles are repelled by the bottom electrode, and they are hurled into the adhesively coated backing where they are retained, standing on end, uniformly spaced from each other. When the electrocoated backing leaves the electrodes, the particles are securely anchored in the adhesive. The material is then looped around the machine

and led into a dryer for curing from where it is taken down and rolled up after the cure is completed.

The force acting on particles in a homogeneous electrostatic field is a function of the field intensity E and the particle charge ne, or

$$F = E.ne \qquad (1)$$

The field intensity E depends primarily on the voltage between the coating electrodes. The particle charge ne depends on many factors, such as dielectric constant, the shape and size of particles, temperature, humidity, conductivity over the particle surface, conductivity through the particle or fiber, premature discharge through sharp points, instantaneous field strength when the fiber touches the lower electrode, average field strength while the particle or fiber is charged by induction and while it is floating in between the electrodes, direction of field and electrode polarity, surface charge of electrodes, frequency or the number of cycles or the number of impulses of a variable field, the contact time of the fiber with the lower electrode and the instant of contact of the fiber or particle with the lower electrode in relation to the instantaneous field strength.

Alternating current is preferred to direct current as a source of supply to coating electrodes when coating cotton, rayon or similar fibers. It results in higher densities and more uniform fiber deposition. By subjecting a particle or fiber to an alternating or variable field of a certain frequency, for instance, to a sinusoidal alternating field of a definite frequency, it is possible to move the fiber in step with the cycle if the fiber characteristics are right. When fibers or particles move in step with the alternating field, the best and most dense electrocoating results. In other words, there is a definite relationship between fiber size and fiber characteristics and the frequency of the electrode potential, or the cycle of the field intensity.

FIBER PREPARATION

To produce fibers which have suitable characteristics for electrocoating, is one of the problems which must be solved by the textile chemists. The cutting, dyeing, bleaching and drying of fibers must be done in such a way that the required characteristics for electrocoating are not impaired. Dyestuffs and dye methods must be adjusted to maintain these fiber characteristics, and the moisture content of fibers, for instance, must be under accurate control. The utmost care must be taken in the fiber preparation; otherwise it is impossible to produce a fabric of uniform density. Even slight variations in density are very undesirable. The wear of fabrics is approximately proportional to the fiber density. With rayon fibers of 3.5 denier, it is possible to obtain by electrocoating, fiber densities of 250,000 to 300,000 fibers per square inch. No woven fabrics can be made as dense, which is one reason why electrocoated fabrics outwear the woven ones by a surprising margin. Wear tests on standard textile testing machines show ratios of 1 to 3 in favor of electrocoated fabrics.

Fabrics of uneven densities would be hard to match for color. The fibers are first cut to even length on a special Behr-Manning cutter. They are then dyed, bleached or processed, before they are coated and the fabrics are not treated after the electrocoating. The color matching must be done with the loose fibers. Variations in the density of the finished fabric would, therefore, show up in color differences.

ADHESIVES

In the electrostatic deposition of fibers, serious consideration must be given to the viscosity of the adhesive so that the required penetration will result from the velocity which the particles attain under the influence of the field. Where this velocity is great, as with particles of small mass under the influence of a powerful field, the viscosity can be at its practical maximum. Any sacrifice of this velocity, such as by increasing the mass of the particles or by field characteristics that are not ideal, must be compensated for by a reduction in viscosity of the adhesive, in order that the required penetration will result. This is probably the basic requirement for an adhesive for electrocoated pile fabric and any adhesive can be used provided the viscosity of its solution or dispersion is controllable. It is obvious that such materials as rubber and its synthetic cousins, reclaims, resins, oils or combinations, are adaptable to this process in the form of solutions or dispersions. Where a final flexible product is required, the use of rubber is naturally indicated. Whether this will be used in the form of latex dispersions or as solvent cements depends largely on the type of fibers used. The ageing of the rubber compound must be sufficient to retain the fiber anchorage throughout the life of the product under the service conditions. While this would have been a rather large order some years ago, it is fortunate for the electrostatic process that rubber compound technique has advanced to the point where such ageing is possible with a good factor of safety in such large volume products as automotive fabrics. Also in the case of such products as carpets, where great flexibility is not required, the advent of the electrostatic process is most opportune. Figure 3 shows an enlarged cross-section through a piece of electrocoated material.

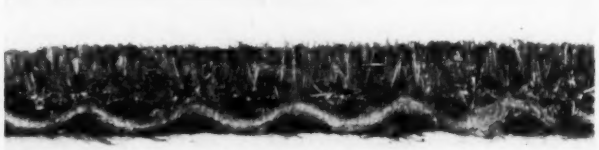


Fig. 3—Enlarged cross-section of electrocoated fabric.

The fibers which happen to be cattle hair are deposited electrostatically and they are held onto the backing with rubber cement. The curing of latex and rubber cement

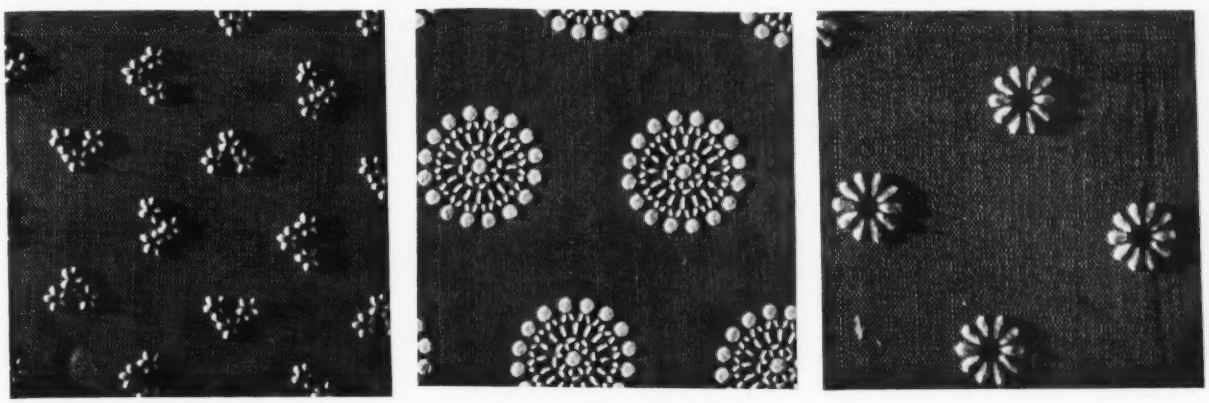


Fig. 4—Patterns which can be produced by the electrocoating Process; all fibers standing on end and uniformly spaced.

is critical and must be under accurate control. Each electrocoating project presents its own adhesive problem which must be solved before successful electrocoating is possible.

Attractive materials can be produced in the dress goods line where cotton or rayon fibers are cut to even length and deposited electrically onto a printed or dyed light voile. Designs are first printed onto the backing with a suitable adhesive. They are then electrocoated with fibers and resemble in their final form fine embroidery. The fibers are uniformly spaced; designs are fluffy and deep. The shadows in Fig. 4 indicate their depth. Some patterns which can be produced are illustrated.

Electrocoated fabrics should find quite a field in the automotive industry for upholstery and sidewall material. There are favorable indications that the shoe and pocketbook trade can use electrocoated fabrics. They should also be used extensively for rugs and carpets, draperies or other decorative materials.

Many interesting problems are left to be solved by scientists and engineers and much work must be done to clear up the laws and relationship of particle and fiber motion in the electrostatic field. Much work is left to be done on suitable adhesives and the fiber preparation. All these problems should be extremely interesting to the textile chemist and colorist.

Reception and Information Committee

Referring to the announcement on page P480, September 15th issue, Harold P. Goller is with the Clinton Co., Greenville, S. C., instead of with the Seydel Chemical Co., as published.

R. H. Smith of the Melrose Hosiery Mills, High Point, N. C., will also assist as a member of the committee on Reception and Information.

Twenty-first

ANNUAL MEETING AND CONVENTION

—RESERVATIONS—

If you have not made your reservations for rooms and sport activities at Pinehurst, **DO IT NOW** through—

D. Stewart Quern
Chairman, Reservation Committee
1930 Harris Road
Charlotte, N. C.

See issue of August 18th
AMERICAN DYESTUFF REPORTER
for hotel rates.

PINEHURST, N. C.
OCT. 31 — NOV. 1

CALENDAR OF COMING EVENTS

Meeting, Mid-West Section, Chicago Towers, October 11, 1941. A full program is scheduled for this meeting.

Meeting, New York Section, Chemists' Club, New York City, October 17th, 1941.

Annual Meeting and Convention, Pinehurst, N. C., October 31st-November 1st, 1941.

Meeting, Southeastern Section, Atlanta, Ga., December 6, 1941.

ANNUAL MEETING AND CONVENTION

R. D. Howerton
Chairman Registration Committee

R. D. Howerton of the Calco Chemical Company, and Treasurer of the Piedmont Section will have the following assistants to help you register on arrival at Pinehurst, October 31st and November 1st:

Jack B. Button, American Cyanamid & Chemical Corp.; Henry B. Dixon, May Hosiery Mills; Leland Atkins, Southern Dyestuff Corporation; J. E. MacDougall, Smith Drum & Company; P. H. Del Plaine, Rohm & Haas, Inc.; Chester L. Eddy, Arnold Hoffman & Company; Chester M. Robbins, Aridye Corporation and Minor C. Hunter, Calco Chemical Company.

Registration fees:

Ladies	$3.00
Members	4.00
Non-members	5.00

(These rates may be slightly reduced if budget permits.)

It is very important for you to register with the Registration Committee as soon as possible after your arrival at Pinehurst. Early registration will enable the Registration Committee to better provide for your meals and banquet accommodations and allow you to get the reduced golf green fees of $1.50 per day. Those playing golf without registration tickets will be charged the regular green fees at Pinehurst, which are higher than the special rate for those registered for the convention.

Admittance to the technical sessions will be restricted to those who have registered. Your registration badge will permit you to attend technical sessions.

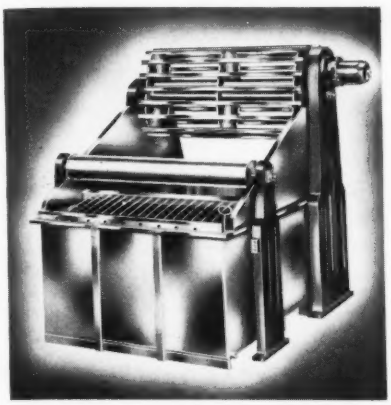
The Registration Desk officially opens at 9:00 Friday morning.

OCTOBER 31st — NOVEMBER 1st
THE CAROLINA, PINEHURST, N. C.

TRADE NOTES • NEW PRODUCTS

• BLICKMAN FOLDER

S. Blickman, Inc., Weehawken, N. J., has recently issued a folder entitled "3 for 5" which features the Blickman Stainless Steel Dye Box. Advantages claimed for this dyeing machine include: handling



Stainless Steel Dye Box.

of more business with fewer dye boxes, lower dyeing costs, reduction in fabric waste, equipment is permanent, production of a better job. Copies of the folder are available upon request to the Blickman company.

• GEIGY RELEASE

Geigy Co., Inc., 89 Barclay Street, New York, N. Y., has recently released circulars describing the following products.

Solophenyl Brown R L—a new direct brown which is said to have exceptionally good fastness to light. It yields a true reddish shade of brown. It is stated that it has good exhaustive properties, even at temperatures as low as 160° F., and may be used in combination with other Solophenyl colors to obtain a variety of shades with excellent resistance to light. It is expected that the product will find considerable use in the dyeing of draperies, automobile and upholstery fabrics or wherever extreme fastness to light is desired. It is also said to possess good fastness to washing (with or without an aftertreatment with formaldehyde) especially when applied to viscose rayon materials.

Solophenyl Grey 4G L—a new homogeneous direct dyestuff said to possess exceptionally good resistance to light. It produces neutral shades of grey. It may be used as a self shade or in combination with other Solophenyl colors for producing fast to light mode shades. It is further said that this dyestuff has unusually good fastness to washing (with or without an aftertreatment of formaldehyde) and should therefore be useful in dyeing spun rayon piece goods. It leaves acetate effects clean enough for most purposes. It is stated that this dyestuff should prove particularly suitable for the dyeing of draperies, furniture materials, and automobile fabrics where the highest fastness to light is required.

• TEXTILE RESEARCH PROGRAM

The first of a series of conferences to explore the most promising fields for textile research, has recently been held at the Hotel Roosevelt, New York, under the auspices of the U. S. Institute for Textile Research. A group of research men from the industry conferred with the steering committee of the Institute. Similar meetings will be held in the future, with other groups from the industry.

Two main problems were discussed: (1) The broad program of the Institute and methods by means of which it could be made most effective; (2) Specific problems which the research men present thought should be the subjects of actual investigation. Complete frankness characterized the meeting, with no holds barred.

Out of this and subsequent conferences, there will be developed the long-range research plan to be followed by the U. S. Institute for Textile Research, in its new working arrangement with the Textile Foundation. This plan will be presented at the annual meeting of the Institute in November.

Representing the U. S. Institute, at the conference were Dr. Harold DeWitt Smith, of A. M. Tenney Associates, treasurer; Dr. W. D. Appel, of the National Bureau of Standards, chairman of the Research Council; Douglas G. Woolf, editor of Textile World, a member of the steering committee of the Institute; and Edward T. Pickard, executive secretary.

Research men from the industry included: A. G. Ashcroft, Alexander Smith & Sons Carpet Co.; Dr. F. Bonnet, American Viscose Corp.; Perrin Collier, Callaway Mills; Duncan Ferguson, Shelton Looms; Ephraim Freedman, R. H. Macy & Co.; M. A. Goldman, Fibre Products Laboratory; Dr. C. J. Huber, U. S. Testing Co.; William O. Jelleme, Pacific Mills; J. E. Phragmen, United Merchants Corp.; Dr. D. H. Powers, Rohm & Haas Co.; W. B. Sellers, Burlington Mills Laboratory; Herbert Schwarz, Wellington Sears Co.; Dr. H. W. Stiegler, American Cyanamid Co.; Werner von Bergen, Forstmann Woolen Co.; Dr. A. C. Walker, Bell Tel. Labs.; Dr. Roy Kienle, Calco Chemical Co.

• GENERAL RELEASES

General Dyestuff Corp., 435 Hudson Street, New York, N. Y., has recently issued circulars describing the following products:

Acid Alizarine Bordeaux B L T—a new homogeneous chrome color. It is said to be well suited for producing deep shades of bluish red on the various forms of wool: loose wool, slubbing, piece goods and yarn. It can be applied by either the afterchrome method or the chrome mordant method and produces dyeings said to be of good fastness to light and very good fastness to washing, fulling, water, sea water and perspiration. The dyeings obtained on a chrome mordant are slightly brighter and bluer than those obtained by the afterchrome process. Circular G-290.

Fast Sulphon Black A—an acid dyestuff which produces greenish shades of black said to possess good fastness to crocking. It is largely used for dyeing hosiery yarns and linings, either alone or in combination with other acid blacks. Circular G-285.

Para Brown G K A—a direct dyestuff which is said to produce attractive shades of yellow brown when coupled with Nitrazol C F Extra. It is particularly well suited for the dyeing of cotton or rayon piece goods. The dyeings are said to show good fastness to washing, water, perspiration and hot pressing. It can be discharged to a good white with either a neutral or an alkaline Rongalite C Paste. It is stated that the dyeings form an excellent ground for white or colored discharge styles which must withstand laundering.

• ARMY TENTS

More than 36,000,000 yards of duck material is being dyed and finished for the Quartermaster Corps as part of the 1942 Army tent program, the War Department reported recently. Cloth is being furnished by the Quartermaster Corps to the successful bidders on the project which involves a new formula recently adopted for use by the Army.

In the dyeing and finishing process the tentage is colored a dark olive drab which blends with trees and foliage thereby making detection from the air more difficult. In addition it receives the customary waterproofing given all Army tentage, and also is treated with a preservative material that protects the tent against mildew, decay, rotting and the ravages of weather, as well as with a fire-resistant fluid that affords protection from flames. The treatment is especially effective against flying sparks which frequently get an ordinary tent ablaze before the fire is discovered.

The total estimated expenditure involved

is approximately $3,995,154, including transportation. Deliveries will be spaced over 30-day periods and will be completed not later than May 31, 1942.

The amount of duck involved is in addition to 1,593,000 yards that is being used for haversacks, carriers, packs, bags, etc., which does not require the special treatment accorded canvas used for tents.

• NEW DEFENSE COURSE

E. W. Ruggles, director of the defense courses conducted at North Carolina State College under the supervision of the United States Office of Education, has announced that the third course in Fabric Testing and Inspection will open on October 1 and run for twelve weeks. All expenses except board, room rent and books are paid by the Federal Government.

Students who enroll in this course will be taught elementary textile design, fabric analysis, fabric calculations, the care and operation of fabric testing equipment, physical and chemical tests for the identification of textile fibers, as well as a study of yarn and fabric defects.

Mr. Ruggles says that this is an excellent opportunity for high school graduates who are now working in textile mills to lay the foundation for future advancement in textile mills, and possibly to qualify for government service in the Quartermaster Corps as fabric inspectors.

• NEW STORAGE TANKS

Erection of eight storage tanks, adding nearly 250,000 gallons to the bulk storage capacity at the Harrison, N. J., plant, has recently been announced by Charles P. Gulick, president and chairman of the board of the National Oil Products Company.

Construction of the 50-foot tanks alongside the firm's facilities fronting on the Passaic River was begun several weeks ago and rushed to completion, Mr. Gulick said. The eight new tanks give the Harrison plant total bulk storage of approximately 3,000,000 gallons.

• TECHNICAL NOTES FROM FOREIGN SOURCES •

The Uses of Natural and Synthetic Rubber in the Wet Processing of Textiles

R. A. Hodgson—J. Soc. Dyers & Col., 56-377, *Sept.*, 1940.—The author in this paper undertakes to outline the chief points of interest in the manufacture of rubber articles which find application in the textile industry, to show how they have been developed to meet specific needs of the industry and to discuss differences which arise when synthetic products are used.

Raw rubber has a very limited application industrially, and practically all that is used comes under the heading of compounded vulcanized rubber. Perhaps the most important single property of rubber is that it can be vulcanized. The exact nature of the phenomenon involved in this process is obscure and a satisfactory definition is lacking, but it has been stated that "In its broadest sense, vulcanization to produce soft rubber is any treatment which decreases the plasticity of the rubber while maintaining its elasticity." The changes which result from this process are caused by the conversion of an elastic thermoplastic substance of low tensile strength into a considerably more elastic, non-thermoplastic substance of greatly increased strength; at the same time other physical and chemical changes accompany vulcanization. The chief and practically universal vulcanizing agent is sulfur, which is added in small or large proportions to the rubber hydrocarbon. It is supposed to react with the rubber molecule at the double bonds to form progressive sulfur bridges between the highly polymerized molecules. If all the double bonds are saturated, hard rubber results, i.e., a compound with almost complete loss of elasticity and improvement in chemical inertness.

The compounding of rubber means the modification of the properties of the vulcanizate to any desired degree. The quality and durability of modern rubber goods may be said to be due to the suitability of the original mixture to fulfill the conditions that it shall (a) process satisfactorily, (b) vulcanize correctly and (c) exhibit, after vulcanization, the specific properties desired by the user. If suitable ingredients are added to rubber before vulcanization it is possible to manufacture products having different properties, e.g., elastic or plastic, hard or soft, solid or porous, etc. Such modified products are obtained chiefly by variations in the method of vulcanization and in the ratio of sulfur to rubber in the vulcanizate, together with the incorporation into the rubber of the following classes of ingredients: (1) Reinforcing agents and stiffeners; (2) plasticizers; (3) accelerators of vulcanization; (4) antioxidants or ageing retarders; and (5) inert fillers or diluents.

These materials are incorporated into the rubber either on external or internal mixers. When the mix is complete, the product is a homogeneous thermoplastic mass which, in most cases, can be sheeted out to a desired thickness on a calender, extruded through an extruder or made up with a solvent to form a dough for spreading. Heat vulcanization is usually carried out in pans which can be heated with direct steam, in hot air or in presses. Moulded rubbers can be vulcanized in all three ways.

Synthetic rubber cannot be defined in chemical terms since it is not applied in the classical sense, which would limit it to an artificially prepared chemical duplicate of natural rubber. A generally recognized definition of synthetic rubber is "those organic substances which possess the property of forcibly retracting to their original size and shape, approximately, after being greatly distorted, e.g., being stretched x per cent of their original length." Commercially, the most important are Neoprene, which is a polymer of 2-chlorobutadiene, the Thiokols, which are condensates of ethylene chloride with sodium polysulfides, and the Buna rubbers, which are interpolymers of butadiene with a proportion of other polymerizable substance, e.g., acrylic nitrile or stryene.

Neoprene can be processed upon standard rubber machinery, the mill, calender, extruder and spreader. Many of the compounding substances suitable for rubber are also applicable to Neoprene, one important difference being that a metallic oxide, such as litharge or zinc oxide, is

essential for vulcanization, and sulfur is actually only a secondary vulcanizing agent. Neoprene cannot be made into a dough with petroleum naphtha; aromatic solvents are necessary for this purpose. Thiokols can be compounded with the majority of the usual ingredients, but vulcanization is brought about by addition of zinc oxide followed by heating; sulfur is of no assistance. The Bunas and Perbunans are processed on standard machinery, but, since they are not so readily plasticized nor so thermoplastic as natural rubber, it is necessary to add large amounts of softeners to the mill before incorporating the other ingredients. Vulcanization is brought about by the use of sulfur and accelerators in a similar manner to the practice used for natural rubber.

These synthetic rubbers all have properties akin to natural rubber, but also possess characteristics of their own which make them superior for certain purposes, notably in their resistance to oxidation and to oils. Neoprene stands up to heat much better than does natural rubber, and has greater resistance to oils and solvents. The Thiokols after vulcanization have not such a high tensile strength and elongation at break as have vulcanized Neoprene or Perbunan mixes, but possess a very high degree of resistance to oils, while their resistance to solvents, especially of the aromatic type, is much superior. Perbunan compounds after vulcanizing exhibit greater resistance to all forms of ageing than does natural rubber, as well as better resistance to abrasion and to flex-cracking.

Industrial application of the synthetic rubbers has in general been restricted to the minority of articles where their superior characteristics justify the additional cost. The new products do not constitute a direct challenge to plantation rubber, but are rather complementary to it, and have widened the scope of the manufacturer in regard to equipment for the chemical and engineering industries. Neoprene, for example, can be compounded with certain ebonites to yield a flexible type of ebonite.

The author considers at some length the present uses of rubber, both natural and synthetic, in various phases of the textile industry. The discussion includes employment of ebonite in pipes and fittings, rubber and ebonite linings for vessels and containers, rubber for covering rollers, the increasing use of Neoprene washpad blankets in calico printing, rubber in belting, gloves and acid-proof hose, as well as the development of "No-Stat" rubber for use in rollers and belting in cases where difficulties arise through the accumulation of static electricity.

The paper is presented by permission of the management of the Dunlop Rubber Co., Ltd., of Manchester.

Raw Materials for the Production of Nylon

J. Soc. Dyers & Col., 56-442, *Oct.*, 1940.—Nylons may be defined as the resinous condensates produced by the interaction of aliphatic dicarboxylic acids with aliphatic diamines at fairly high temperatures and, obviously, it is possible to make a large number of such products of varying physical properties. Only a few dibasic acids and diamines, however, are suitable for the manufacture of nylons adapted for the production of bristles and textile fibers. Considerations of quality and price appear to indicate that the most satisfactory raw materials in these fields are adipic and sebacic acids and hexa- and deca-methylenediamines.

Adipic acid is produced by the oxidation of hexahydrophenol, whereas methyladipic acid results when methylhexahydrophenol is oxidized. Since hexahydrophenol can also be made from *cyclo*hexene, which is readily obtainable from suitable petroleum fractions, there is justification for assuming that costs of manufacture will be gradually reduced.

Sebacic acid in good yield and high purity is manufactured by the high pressure treatment of castor oil with caustic soda (U. S. P. 2,182,056).

High yields of hexamethylenediamine, white solid, M.P. 39° C., are obtained by the high pressure catalytic hydrogenation of adiponitrile, a compound which is produced by passing a mixture of ammonia and adipic acid vapor over a dehydration catalyst at high temperature. Hexamethylenediamine can also be made by the high pressure catalytic hydrogenation of epsilon-caprolactam. The latter compound is made from heptaldehyde which, itself, is derived from castor oil. Decamethylenediamine, white solid, M.P. 60° C., is obtained *via* sebacic acid and sebaconitrile.

Effect of the Sulfuryl Chloride Process on Wool Dyes

J. Soc. Dyers & Col., 56-443, *Oct.*, 1940.—Reference has been made in these columns (AMERICAN DYESTUFF REPORTER, 29-7, April 1, 1940) to the sulfuryl chloride process for making wool unshrinkable. A technical circular issued by the Imperial Chemical Industries, Ltd., provides information as regards the effect of this process on the shade and fastness of that company's acid and acid mordant dyes.

The change of shade, washing and light fastnesses, and in some cases milling fastness, in comparison with dyeings on untreated wool, are recorded in tabular form. The results show that shades on wool previously treated with sulfuryl chloride are, in many instances, slightly different from those on normal wool, but in most cases there is comparatively little difference in the washing fastness. When the sulfuryl chloride treatment is applied to previously dyed wool, however, many of the dyeings suffer a serious change in shade or loss in strength, or both, and the fastness to washing, except of the mordant dyes, is seriously impaired.

Of the acid dyes, the Carbolan and Coomassie types are the most resistant to the process. It is recommended, therefore, that, where possible, treatment should precede dyeing, since there is then a larger range of satisfactory dyes available. In those cases where a particular shade or fastness property is of prime importance, customers are recommended to make tests on their own material under their own conditions.

MILDEW-RESISTANT TREATMENTS

(Continued from Page 504)

treated fabric, as well as on the untreated sterilized controls.

RESULTS

Table 1 gives results showing the effect on the fabric of various mildew-preventing treatments applied to unbleached cotton duck and the effectiveness of these treatments in preventing mildew growth. Each value is calculated from the average of 10 breaking strength determinations and is reported as the percentage change in breaking strength of the treated fabric and of the treated fabric after inoculation and incubation with *Chaetomium globosum*. Since the change in strength in both cases is expressed in terms of the untreated fabric, these values therefore represent the change due to the chemical treatment itself and the change due to the combined effect of treatment and mildew growth.

The table also gives the change in strength due only to the mildew growth. This was obtained as the percentage change in breaking strength of the treated fabric after inoculation and incubation and is expressed in terms of the treated fabric. This value shows merely the effectiveness of the treatment in preventing mildew growth and does not include the deteriorating or strengthening effect on the fabric that the treatment may have. The percentage gain in weight reported in the table and due to the treatment is calculated from the weights on 20 strips.

In order to determine for each treatment whether the change is a significant gain or loss these data were analyzed statistically by the "*t*" test. It was found that percentage variations in breaking strength of 8 per cent and greater are significant.

In interpreting the results that indicate the effectiveness of the treatments as mildew-resisting agents it should be pointed out that the laboratory test to which the samples are subjected is very severe. In the test method the temperature and moisture conditions are ideal for growth of the organism. The test greatly accelerates and exaggerates the deterioration encountered in the normal use of fabrics for shower curtains, porch furniture upholstery, and even tents or awnings. For all practical purposes, therefore, a reported change in breaking strength due to mildew growth even as large as 10 per cent has little or no importance. And perhaps a treatment allowing as much as 20 or 25 per cent loss in strength might still be satisfactory under many conditions of use.

For convenience in reporting the results the treatments are arranged into 3 groups according to the chief constituent used in the treatment: 1, organic compounds; 2, organometallic compounds and metallic salts of organic compounds; and 3, inorganic compounds.

The treatments in the organic group include an acetylating mixture, a resin, chlorothymol and thymol with phenyl salicylate, cutch, two chlorine substituted phenols and a sodium salt of one of them used alone and combined with other compounds, salicylanilide, and three quaternary ammonium compounds.

As tested by the method employed in this study the acetylation process using acetic anhydride, glacial acetic acid, and zinc chloride for 20 hours at 20°-25° C. gives very good protection against mildew. The temperature and concentration of the acetylating mixture must be very carefully and accurately controlled or the reaction becomes too rapid and the fabric is stiffened and deteriorated. As shown in the previous study, shorter periods of acetylation are not sufficient to give mildew protection to the fabric and if acetylated for longer than 20 or 21 hours the fabric loses much strength. This loss, however, is chiefly due to the deteriorating effect of the treatment and not to mildew growth.

The resin formed with acetone and formalin can not be relied on to give protection against mildew. Much difficulty was encountered in getting this treatment applied evenly and uniformly on the fabric strips. This is evidenced by the fact that the increase in weight of the strips due to the treatment ranged from zero to 7.5 per cent. The strips having only a small amount of resin deposited on them allowed considerable mildew growth and consequently had low breaking strength values while the strips with large amounts of resin were well protected against mildew and had correspondingly high values.

Since chlorothymol and related compounds are volatile in steam, steam sterilization of such treated fabrics often removes the special finish. But if not sterilized chlorothymol gives good protection against *Chaetomium globosum* even with a very small quantity held on the fabric. In ordinary use, however, chlorothymol-processed fabrics need not be subjected to steam.

When chlorothymol is refluxed for four hours with morpholine and sodium carbonate, an oil is formed which yields crystals melting at 54° C. This product dissolved in alcohol has very satisfactory mildew-resisting action when applied to cotton. However about a 15 per cent solution must be used to give protection.

When thymol is combined with phenyl salicylate, mixed crystals are obtained which can be used as a mildew-preventing agent. However this treatment permits about 10 per cnt loss in strength even with large amounts held in the fabric. Steam sterilization does not remove this finish from the fabric as in the cases of thymol and chlorothymol used alone.

Of the treatments using cutch or catechu, method 1 with copper sulfate and ammonium hydroxide is easier to apply, gives more uniform results and requires less cutch to give protection against mildew than method 2, with potassium dichromate. In this latter method enough

cutch is not always held on the fabric to be effective. The amount, 2.7 per cent on the fabric, is not sufficient for protection since it allowed great loss in strength. However, as shown in a previous study[2], 3.7 per cent gives excellent protection but it makes the fabric very stiff. These cutch treatments dye the fabric a dark brown.

The chlorinated phenols, 2-chloro-o-phenylphenol and pentachlorophenol, are good mildew-resisting agents. The salt, sodium pentachlorophenolate, also gives good protection but is very soluble and tends to rinse out of the fabric. If the fabric is treated first with cadmium chloride and then with sodium pentachlorophenolate the insoluble pentachlorophenolate is formed and held between the fibers and yarns. This two-bath process seems to strengthen the fabric and is entirely satisfactory in inhibiting *Chaetomium globosum*. However, because of the pungent and irritating odor of these phenols and their salts, fabrics treated with them are very difficult to handle.

Similarly to chlorothymol, both 2-chloro-o-phenylphenol and sodium pentachlorophenolate also may be condensed with morpholine to form satisfactory mildew-preventing agents. The phenol yields a condensation product melting higher than 300° C. Since it is soluble in hot water and practically insoluble in cold, it is held in the fabric. The product formed from sodium pentachlorophenolate and morpholine melts at about 120° C. It gives excellent protection against mildew and does not rinse out of the fabric. Also the treated strips are free from the sharp irritating odor of pentachlorophenol.

Salicylanilide gives excellent protection against mildew provided the treated fabric is not steam sterilized. On the other hand when salicylanilide is applied with a water-repelling agent of wax and aluminum acetate, it gives complete protection even after sterilization. Steaming, however, is not necessary in practical applications of salicylanilide as a mildew-resisting agent.

According to the results of this study two of the quaternary ammonium compounds tried, an alkylated dimethyl benzyl ammonium phosphate and a chloride, are entirely satisfactory in inhibiting *Chaetomium globosum*. Both have been used commercially for some time. The tetra-alkyl ammonium halide has a deteriorating action on the fabric and gives practically no protection against mildew.

The treatments grouped under the second heading, organometallic compounds and metallic salts of organic compounds, include a copper acetonate, a mercury salicylate and oleate, zinc and copper naphthenates, cadmium and copper soaps, and morpholine and hydroxyquinoline condensation products.

Copper propionyl acetonate, p-tolyl mercury salicylate and phenyl mercury oleate are all used commercially and resist mildew effectively. The copper compound colors the fabric gray-green. Information regarding the toxicity of the mercury compounds is not available.

As indicated in the table, copper and zinc naphthenate applied either in water or in Stoddard solvent give complete mildew-resisting protection. Copper naphthenate is green, zinc naphthenate is colorless; both have a pungent disagreeable odor and stiffen the fabric slightly. In a few instances the treated fabric was even stronger than the untreated.

Two-bath processes of soap with cadmium chloride and copper sulfate form insoluble soaps in the fabric. The cadmium soap treatment is colorless and gives excellent protection against mildew. The copper soap is fairly good although even with a 10 per cent solution of copper sulfate a slight loss in strength was observed; whereas a 1 per cent solution of cadmium chloride completely protects the fabrics. These soap treatments have an advantage over many of the others in that they are easy to apply.

Morpholine combines with inorganic salts to form an insoluble product held directly in the fabric. With cadmium chloride and with copper sulfate, excellent protection against mildew is obtained. The copper sulfate combination colors fabric blue but the cadmium chloride one is colorless. These products were developed in this laboratory and a public service patent, No. 2,247,339, covering the treatment of fabric with morpholine and inorganic salts has been granted to the junior author.

The chemical, 8-hydroxy quinoline, also combines with inorganic salts to form insoluble mildew-preventing agents held directly in the fabric. The aluminum compound is bright yellow colored on the fabric and the magnesium one is dull yellow. According to the conditions of this test both are very effective in protecting against mildew.

Of the finishing treatments using inorganic salts, the only one which is entirely satisfactory for inhibiting mildew growth is cadmium chloride followed by borax. It gives excellent protection. Chromium fluoride allows almost complete breakdown in fabric strength under the severe conditions of the test used although previous trials indicated that it had some value as a mildew preventive. The two-bath process of copper sulfate and sodium carbonate gives mildew protection but the treatment itself has a slight deteriorating action on the fabric.

SUMMARY

Thirty-two different finishing treatments were applied to a degreased and desized unbleached cotton duck. Only one or two treatments had a deteriorating action on the fabric; a few seemed to strengthen it. The mildew resistance of the treated fabric was measured by its loss in breaking strength after inoculation and incubation with the test organism, *Chaetomium globosum*.

After testing with *Chaetomium globosum* the following 28 treatments allowed little or no loss in fabric strength:

Acetylation.
Chlorothymol not steam sterilized.
Condensation product of chlorothymol and morpholine.
Thymol with phenyl salicylate.
Cutch.

Mildew-resistant Treatments

Finishing Treatment	Treatment Bath 1 — Chemical	Concentration (Grams per 100 ml. Water)	Temperature at Which Fabric Was Immersed (°C.)
Organic compounds:			
Acetic anhydride and glacial acetic acid	Acetic anhydride	33.3 [3]	20–25
	Glacial acetic acid	27.0	
	Zinc chloride	4.7	
Acetone with formalin	Acetone	8.0	50–60[4]
	Formalin	39.0	
	Soda ash	2.0	
Chlorothymol	Chlorothymol	2.0[6]	25–30
Chlorothymol	Chlorothymol	2.0[6]	25–30
Chlorothymol with morpholine	Chlorothymol with morpholine[7]	3.0[6]	25–30
Chlorothymol with morpholine	Chlorothymol with morpholine[7]	15.0[6]	25–30
Thymol with phenyl salicylate	Thymol with phenyl salicylate[8]	40.0[6]	25–30
Thymol with phenyl salicylate	Thymol with phenyl salicylate[8]	40.0[6]	25–30
Cutch, Method 1	Cutch	0.3	100
Cutch, Method 2	Cutch	1.3	100
2-Chloro-o-phenylphenol	2-chloro-o-phenylphenol	3.0[10]	25–30
2-Chloro-o-phenylphenol with morpholine	2-chloro-o-phenylphenol with morpholine[11]	3.0	80–85
Pentachlorophenol	Pentachlorophenol	3.0[10]	25–30
Sodium pentachlorophenolate	Sodium pentachlorophenolate	2.0	25–30
Sodium pentachlorophenolate with cadmium chloride	Cadmium chloride	5.0	100
Sodium pentachlorophenolate with morpholine	Sodium pentachlorophenolate with morpholine[12]	3.0[12]	25–30
Sodium pentachlorophenolate with morpholine	Sodium pentachlorophenolate with morpholine[12]	3.0[12]	25–30
Salicylanilide	Salicylanilide	0.2	25–30
Salicylanilide	Salicylanilide	0.2	25–30
Salicylanilide with wax and aluminum acetate emulsion	Salicylanilide emulsion	0.2	80–85
	Wax and aluminum acetate emulsion	3.0	
Quaternary ammonium compounds:			
Tetra-alkyl ammonium halide	Tetra-alkyl ammonium halide	5.0	43[4]
	50% ethyl alcohol	10.0	
Alkyl dimethyl benzyl ammonium phosphate	Alkyl dimethyl benzyl ammonium phosphate	7.0	25–30
Alkyl dimethyl benzyl ammonium chloride	Alkyl dimethyl benzyl ammonium chloride	0.1	25–30
Organometallic compounds and metallic salts of organic acids:			
Copper propionyl acetonate	Copper propionyl acetonate	3.0[14]	25–30
Paratolyl mercury salicylate	Paratolyl mercury salicylate	10.0[15]	25–30
Phenyl mercury oleate	Phenyl mercury oleate[16]	0.8	25–30
Phenyl mercury oleate	Phenyl mercury oleate[16]	2.0	25–30
Phenyl mercury oleate	Phenyl mercury oleate[16]	0.25[10]	25–30
Phenyl mercury oleate	Phenyl mercury oleate[16]	0.8[10]	25–30
Copper naphthenate	Copper naphthenate	4.8	25–30
Copper naphthenate	Copper naphthenate	28.5	25–30
Copper naphthenate	Copper naphthenate	33.3[10]	25–30
Zinc naphthenate	Zinc naphthenate	4.8	25–30
Zinc naphthenate	Zinc naphthenate	20.0	25–30
Zinc naphthenate	Zinc naphthenate	20.0[10]	25–30
Zinc naphthenate	Zinc naphthenate	33.3[10]	25–30
Soap, sodium oleate, with cadmium chloride	Soap	1.0	100
Soap, sodium oleate, with copper sulfate	Soap	1.0	100
Morpholine with cadmium chloride	Cadmium chloride	5.0	100
Morpholine with copper sulfate	Copper sulfate	5.0	100
Morpholine with copper sulfate	Copper sulfate	5.0	100
8-Hydroxy quinoline with aluminum acetate	Aluminum acetate	2.0	100
8-Hydroxy quinoline with magnesium chloride	Magnesium chloride	5.0	100
Inorganic compounds:			
Cadmium chloride with borax	Cadmium chloride	5.0	25–30
Chromium fluoride	Chromium fluoride	0.65	90–95
Copper sulfate with sodium carbonate	Copper sulfate	5.0	100

[1]The plus sign preceding the value indicates an increase in breaking strength; a minus sign indicates a decrease. All values of 8 per cent and greater represent a significant change in strength from the original untreated fabric.

[2]The plus sign preceding the value indicates an increase in breaking strength; a minus sign indicates a decrease. All values of 8 per cent and greater represent a significant change in strength from the corresponding treated and uninoculated fabric.

[3]Amount required to process one strip of fabric.

[4]After treatment the fabric is dried and then heated 5 minutes at 120° C.

[5]Treated fabric was not steam sterilized as is usual in the test procedure.

[6]Chemical dissolved in ethyl alcohol.

[7]Crystals, M.P. 54° C., obtained from chlorothymol, 50 gm.; moropholine, 25 ml.; sodium carbonate, 3 gm.; and water, 50 ml., refluxed for 4 hours and the product separated with very dilute hydrochloric acid.

[8]Mixed crystals obtained from thymol, 30 gm.; phenyl salicylate, 10 gm.; and phthalic acid, 0.4 gm.; heated to 37°-40° C.

[9]Add ammonium hydroxide to form cuprammonium salt.

TABLE 1
Unbleached Cotton Duck

Procedure							Change in Breaking Strength of Treated Fabric After Inoculation and Incubation with Chaetomium globosum	
Bath 2								
Time	Chemical	Concentration	Temperature at Which Fabric Was Immersed	Time	Gain in Weight of Fabric Due to Treatment	Change in Breaking Strength of Fabric Due to Treatment[1]	Due to Treatment and Mildew Growth[1]	Due to Mildew Growth[2]
		Grams per 100 ml. Water	°C.	Mins.	%	%	%	%
20–25	20 hrs. ——	—	—	—		—14	— 8	+ 6
50–60[4]	2 min. ——	—	—	—	2.8	—11	—81	—78
25–30	30 min. ——	—	—	—	0	+ 2	—87	—87
25–30	30 min ——	—	—	—	0	+ 9[5]	+ 8[5]	— 0.5[5]
25–30	30 min. ——	—	—	—	1.0		— 2	
25–30	30 min. ——	—	—	—	0.2		—97	
25–30	3-4 min. ——	—	—	—	79	+ 7	— 8	—14
25–30	3-4 min. ——	—	—	—	66	+ 0.5[5]	— 9[5]	—10[5]
100	Overnight { Copper sulfate ... Ammonium hydroxide ... }	1.0 0	25–30	10–15	1.6	— 5	+ 5	+10
100	Overnight Potassium dichromate ...	0.3	90	45	2.7	+ 1	—55	—55
25–30	30 min. ——	—	—	—	1.1	+ 5	+ 7	+ 2
80–85	30 min. ——	—	—	—	—		+12	
25–30	30 min. ——	—	—	—	2.6	+ 0.5	+ 7	+ 7
25–30	30 min. ——	—	—	—	0.4	+ 3	— 3	— 6
100	15 min. Sodium pentachlorophenolate ..	3.0	25–30	15	7.7	+13	+10	— 2
25–30	30 min. ——	—	—	—	4.1	+ 6	+ 5	— 1
25–30	30 min. ——	—	—	—	4.1	+ 9[5]	+ 8[5]	— 1[5]
25–30	30 min. ——	—	—	—	0.1	— 1	—97	—97
25–30	30 min. ——	—	—	—	0.1	— 1[5]	— 8[5]	— 7[5]
80–85	3 min. ——	—	—	—	2.8	— 1	+ 0.5	+ 2
43[3]	1 min. ——	—	—	—	13	—18	—89	—87
25–30	30 min. ——	—	—	—	1.3	+ 5	+ 6	+ 0.5
25–30	30 min. ——	—	—	—	—	+ 0.5	— 4	— 4
25–30	30 min. ——	—	—	—	2.4	+ 3	+ 1	— 3
25–30	30 min. ——	—	—	—	2.0	—	— 6	—
25–30	30 min. ——	—	—	—	1.5	— 6	— 2	+4
25–30	30 min. ——	—	—	—	1.9	— 2	— 2	0
25–30	30 min. ——	—	—	—	0.9	+ 4	—18	—21
25–30	30 min. ——	—	—	—	1.1	+ 4	+ 4	+ 0.5
25–30	30 min. ——	—	—	—	—	+ 2	+ 7	+ 5
25–30	30 min. ——	—	—	—	—	+ 4	+13	+ 9
25–30	30 min. ——	—	—	—	—	+ 7	+12	+ 5
25–30	30 min. ——	—	—	—	—	+ 7	+ 4	— 3
25–30	30 min. ——	—	—	—	16	+13	+13	0
25–30	30 min. ——	—	—	—	—	+12	+ 9	— 3
25–30	30 min. ——	—	—	—	—	+ 6	+ 7	+ 0.5
100	10 min. Cadmium chloride ...	1.0	100	30	0.3	— 4	— 1	+ 3
100	10 min. Copper sulfate ...	10.0	100	30	1.1	— 2	—10	— 8
100	15 min. Morpholine ...	[17]	25–30	15	1.6	+ 3	—4	— 7
100	15 min. Morpholine ...	[17]	25–30	15	1.8	— 1	— 4	— 3
100	15 min. Morpholine ...	[17]	25–30	15	1.5	— 3[5]	— 4[5]	— 1[5]
100	30 min. 8-Hydroxy quinoline ...	2.5[18]	90–95	15	—	— 4	+ 8	+13
100	30 min. 8-Hydroxy quinoline ...	2.5[19]	90–95	15	—	+ 7	+ 8	+ 0.5
25–30	30 min. Borax ...	4.0	25–30	10	3.0	— 2	— 3	— 1
90–95	30 min. ——	—	—	—	0.1	— 2	—84	—84
100	30 min. Sodium carbonate ...	5.0	100	10	1.0	— 9	—10	— 2

[1]Chemical dissolved in Stoddard solvent.
[2]Crystals, M.P > 300° C., obtained from 2-chloro-o-phenylphenol, 5 ml.; morpholine, 25 ml.; sodium carbonate, 5 gm.; and water, 20 ml., refluxed for 7 hours and the product crystallized.
[3]Crystals, M.P. 120° C., obtained from sodium pentachlorophenolate, 50 gm.; morpholine, 25 ml.; sodium carbonate, 3 gm.; and water, 50 ml., refluxed for 4 hours and the product crystallized.
[4]Chemical dissolved in morpholine.
[5]Chemical dissolved in methyl alcohol.
[6]Commercial solution (concentration unknown) dissolved in water.
[7]Commercial solution consisted of 10 per cent emulsion of phenyl mercury oleate in paraffin base oil.
[8]Fabric strip immersed in 50 ml. of morpholine.
[9]Solution made slightly acid with acetic acid.
[10]Solution made slightly alkaline with ammonium hydroxide.

2-chloro-o-phenylphenol.
Condensation product of 2-chloro-o-phenylphenol and morpholine.
Pentachlorophenol.
Sodium pentachlorophenolate.
Sodium pentachlorophenolate followed by cadmium chloride.
Condensation product of sodium pentachlorophenolate and morpholine.
Salicylanilide not steam sterilized.
Salicylanilide with a wax and aluminum acetate emulsion.
An alkyl dimethyl benzyl ammonium phosphate.
An alkyl dimethyl benzyl ammonium chloride.
Copper propionyl acetonate.
P-tolyl mercury salicylate.
Phenyl mercury oleate.
Copper naphthenate.
Zinc naphthenate.
Soap followed by cadmium chloride.
Soap followed by copper sulfate.
Cadmium chloride followed by morpholine.
Copper sulfate followed by morpholine.
Aluminum acetate followed by 8-hydroxy quinoline.
Magnesium chloride followed by 8-hydroxy quinoline.
Cadmium chloride followed by borax.
Copper sulfate followed by sodium carbonate.

Dyes from Citrazinic Acid—

(Concluded from Page 503)

by blocking of the carboxyl group. The good washing fastness of the disazo chrome top colors is noteworthy.

Special attention is called to the yellow dye made from the butylether of citrazinic acid and aniline-2.5-disulfonic acid (No. 22). The shade of this dye is almost identical with Quinoline Yellow and it is a much greener yellow than any we have come across, with the solitary exception of Quinoline Yellow. The citrazinic dye may be manufactured at a cost considerably under that of Quinoline Yellow and has also an enhanced light fastness. The chrome top color is a very bright yellow and should be also of interest. Some of the other dyes enumerated such as those made with citrazinic acid, on one hand, and with picramic acid (No. 7C), o-aminophenol-p-sulfonic acid, (No. 19C), and amino-azo-benzene-p-sulfonic acid (No. 21C) on the other hand, are also very promising as chrome top colors.

It was noticeable that the dyestuffs formed from the monobutyl ether of citrazinic acid had a much more brilliant shades than the dyes produced from the acid itself. A more thorough exploitation of the dyestuff possibilities derived from citrazinic acid and its functional and other derivatives both in the azoic as well as other fields appears to have a good chance to produce commercially interesting products. The industry's attention is called to this new field, in the hope that further work by dyestuff manufacturers may yield results beneficial to both the dye producing and consuming industries.

We express our indebtedness to Charles Pfizer & Co., sponsors of the above work for their permission to release these results.

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OPEN FORUM

55—I am interested in securing the chemical structure of the following dyes listed under Prototype 24, 96, 97 in the 1940 Year Book of the American Association of Textile Chemists and Colorists.

Any information which may be imparted in regard to this will be appreciated.—**J. L. N.**

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